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UNITED STATES
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HAROLD L. ICKES, SECRETARY

BUREAU OF MINES
A. C. FIELDNER, ACTING DIRECTOR

INFORMATION CIRCULAR

ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1939

BY

A. C. FIELDNER AND R. E. BREWER

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ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1939[1]

By Arno C. Fieldner[2] and R. E. Brewer[3]

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[1] The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7105."

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INTRODUCTION

This report is the fourth of a series of annual summaries of research and technologic work on coal by the Technologic Branch of the Bureau of Mines.

The coal research program of the Bureau has been broadened and made more flexible to adapt it to changing conditions and needs of the coal industry, and as a result information as to the properties and composition of coal has been increased and new and improved practices in mining, preparation, and utilization of coal have been devised. The present report is a synopsis of the papers published and of other work in progress during the fiscal year ended June 30, 1939.

ACKNOWLEDGMENT

This report has been prepared from material included in the annual reports submitted by the supervising heads of all sections in the Coal Division and of certain sections in the Mining, Nonmetals, and Explosives Divisions of the Technologic Branch, from information supplied by the Health and Safety Divisions of the Health and Safety Branch, and from recent publications by members of the divisions named. Special acknowledgment is made to the following members of the Bureau of Mines staff:

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Station, College Park, Md.
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Pennsylvania State College, State College, Pa.
State of Illinois Department of Mines and Minerals, Springfield, Ill.
University of Alabama, Tuscaloosa, Ala.
University of North Dakota, Grand Forks, N. Dak.
University of Washington, Seattle, Wash.

The personnel of a number of coal companies gave valuable advice and assistance in connection with various studies and investigations.

PROPERTIES AND COMPOSITION OF AMERICAN COALS

Progressive improvement and development of methods for determining the chemical and physical properties of American coals have made available much valuable information for their more economical preparation and for predicting their suitability for various present and purposed uses.

Some of the outstanding recent achievements in coal research and technology by the Bureau of Mines staff and others have been summarized briefly.4/ Certain standard specifications for and methods of testing coal and its products are admittedly empirical, but it is not to be expected that all of them can or should be free of empirical conditions.5/ Correct interpretation and application of these measures of coal properties to what they may be expected to predict are more important than are the properties themselves in achieving the desired coordination of knowledge on coal among coal investigators and laymen.

4/ Fieldner, A. C., and Rice, W. E., Progress in Coal Research and Technology: Min. Cong. Jour., vol. 25, February 1939, pp. 38-40.

5/ Nicholls, P., Discussion of "Recent Developments in Coal Research" by H. H. Lowry: Proc. 2d Ann. Coal Conf., West Virginia Univ., Tech. Bull. 13, February 1939, pp. 81-82.

Inspection, Sampling, and Analysis

During the fiscal year there was an increased demand from district boards established under the National Bituminous Coal Act, the National Bituminous Coal Commission, Government agencies, and prospective bidders on Government coal contracts for inspection, sampling, and analysis of coal produced at many individual mines.

One new coal-sampling truck was put into service early in the year. This and two other trucks were kept in field service virtually the entire year. Coal was inspected at 14 anthracite breakers, and 99 samples of anthracite were collected in the Pennsylvania anthracite field in connection with awarding contracts for purchases of anthracite required by various departments of the Government. Bituminous-coal inspection and the collection of samples for use of the Government in making coal purchases and for general information required 943 tipple, 13 grindability, and 28 experimental-moisture samples, as follows: 467 tipple and 13 grindability samples at 112 mines in Illinois; 46 tipple samples at 9 mines in Indiana; 72 tipple and 28 experimental-moisture samples at 25 mines in Ohio; 289 tipple samples at 77 mines in eastern Kentucky; 22 samples at 12 mines in Pennsylvania; 39 samples at 8 mines in Virginia; and 8 samples at 2 mines in West Virginia. In cooperation with the Bituminous Coal Producers Board for District No. 2, 58 tipple samples were collected at 13 mines in western Pennsylvania, and with the Board for District No. 9, a total of 262 tipple samples at 72 mines and 40 face samples at 10 mines in western Kentucky. At the request of the National Bituminous Coal Commission, 32 tipple samples were collected at 15 mines in District No. 6 (Panhandle), West Virginia, and 7 samples at 1 mine in Ohio. In connection with the surveys of carbonizing and hydrogenation properties of American coals, 5 face samples, 1 carbonization sample, and 1 sample for microscopic examination were collected at 1 mine in Virginia; 14 face, 3 carbonization, 3 samples for microscopic examination, and 1 hydrogenation sample at 3 mines in West Virginia; and 7 face, 1 carbonization, 1 sample for microscopic examination, and 1 hydrogenation sample at 1 mine in Pennsylvania.

Anthracite and bituminous-coal field work thus collected 1,401 tipple samples at 360 mines, 66 face samples at 15 mines, 13 grindability and 28 experimental-moisture samples at mines included in the above summary, and 5 carbonization samples, 5 samples for microscopic examination, and 2 samples for hydrogenation tests at 5 mines. These represent an increase of more than 100 percent in the number of mines sampled and number of samples collected compared with the previous year.

The Northwest (Seattle, Wash.) Experiment Station and the Oregon Department of Geology and Mineral Industries are investigating the physical and chemical properties of the coals of Oregon. The program includes the collection of samples for chemical analysis, low-temperature carbonization assay and determination of agglutinating value, and slacking and friability indexes of all of the coals now being mined in the State and of accessible deposits not active at present.

Original from THE OHIO STATE UNIVERSITY

During the year, 83 mine samples were taken at 41 mines and prospects and 80 samples of delivered coal were collected at 28 mine tipples in the State of Washington. The standard samples have been analyzed to bring up to date the information in Technical Paper 491, Analyses of Washington Coals.

During the year 10,097 individual samples of coal, coke, and related products were analyzed in the coal-analysis laboratory. Of these, 6,283 were analyzed in connection with fuel-inspection service for purchases of coal by 26 Federal agencies and 1,085 for 6 State institutions; 2,541 related to the research program of the Bureau; and 188 were samples submitted by 7 other United States agencies for checking purposes. The testing program represented an increase of 16.3 percent over the preceding year. Each sample received an average of 12.5 determinations, making approximately 126,200 determinations for the year.

In addition to the analyses made in the coal-analysis laboratory, 355 samples of coal, coal ashes, coal-ash slags and clinkers, fuel-saving compounds, dusts for rock-dusting coal mines, boiler-water compounds, boiler scales, boiler waters, and products of coal hydrogenation were analyzed for special purposes by the miscellaneous analysis section.

Coal-analysis service, especially information on average analyses of coals from given mines for use in connection with awarding and enforcing coal contracts for furnishing coals to various Federal and State institutions, recommendations on proper sampling methods, and advice on changes in specifications for the purchase of coal and on the kinds of coal required for certain types of plants was given to many Federal and State agencies and to the general public.

Tables of typical coal analyses have been compiled for use of the Office of the Consumers Counsel.[6]

The revision of Technical Paper 76, issued in 1938 as Technical Paper 586, Notes on the Sampling and Analysis of Coal, has been reprinted.[7] A revised edition of Technical Paper 8, Methods of Analyzing Coal and Coke[8], and Technical Paper 590, Analyses of Pennsylvania Bituminous Coals, were issued.[9]

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[6] Rice, W. E., and Moran, H. E., Explanation and Method of Compiling of the Tables of Typical Coal Analyses: Pamphlet by Office of the Consumers Counsel, 1940.

[7] Fieldner, A. C., and Selvig, W. A., Notes on the Sampling and Analysis of Coal: Fuel, vol. 18, January-April 1939, pp. 4-13, 56-63, 68-77, 110-115.

[8] Stanton, F. M., Fieldner, A. C., and Selvig, W. A., Methods of Analyzing Coal and Coke: Bureau of Mines Tech. Paper 8, (rev.), 1939, 59 pp.

[9] Ashley, G. H., Toenges, A. L., Anderson, R. L., Rice, W. E., Stull, C. M., Snyder, N. H., Swingle, R. J., Cooper, H. M., and Abernethy, R. F., Analyses of Pennsylvania Bituminous Coals: Bureau of Mines Tech. Paper 590, 1939, 503 pp.

The relationships between heating value, volatile matter, and ash content for 1,008 samples of anthracite have been presented graphically.[10] Oxidation with air at high temperatures shows that heating value, volatile matter, and amount of oxygen consumed change directly with the extent of oxidation, irrespective of the quality of coal. The deviations with temper ture up to 300° C. or less enable the relations to be detected quickly and positively by means of graphs.

Because of the continuing value of information on the spontaneous combustion of coal, Technical Paper 311, Factors in the Spontaneous Combustion of Coal, has been reissued.[11]

Special Physical Testing

Sampling

Principles governing the sampling of coal for ash content and for size consist have been determined. Mathematical derivations necessary for the establishment of rigorous sampling formulas and their practical applications in drafting coherent sampling specifications will be presented in a future publication. Statistical size constants of subdivided materials, w specific application to blasted and pulverized coal, have been determined.

Abrasiveness

An important item in the cost of grinding coal for use as pulverized fuel is that of maintaining the grinding mill, caused by the abrasive wear of its parts by the coal. A method for estimating the abrasiveness of coa has been developed in preliminary form at the Northwest (Seattle, Wash.) Experiment Station. A test disk studded with projections is revolved at constant thrust or bearing pressure against the coal sample confined in a cylindrical steel mortar. A steel ring encircles the test piece and clamp it horizontally to the base of a vertical shaft, thus providing a suitable mounting for the test piece, protecting it from metallic contact with the mortar, and definitely limiting its exposed surface. The loss in weight o the test piece after a definite number of revolutions at constant pressure against the coal sample is the measure of the abrasiveness of the coal. The relative abrasiveness of various coal samples was found to vary considerably, depending essentially on the nature and amount of the extraneous impurities present. The pure-coal (float 1.35 specific gravity) portions of the various samples examined were found to be relatively low in abrasiveness, whereas the impurities of these same samples had an extreme abrasiveness that varied in degree with the nature of the materials.

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[10] Scott, G. S., Jones, G. W., and Cooper, H. M., Effect of Oxidation of Anthracite on Its Heating Value: Ind. and Eng. Chem., vol. 31, 1939, pp. 1025-1027.

[11] Hood, O. P., Spontaneous Combustion of Coal: Bureau of Mines Inf. Ci 7074, 1939, 5 pp.

[12] Roller, P. S., Statistical Size Constants of Subdivided Materials, With Specific Application to Blasted and Pulverized Coal: Lecture at 501st meeting of American Chemical Society of Washington, May 12, 193 Washington, D. C.

Relation Between Physical and Chemical Properties

The relationships between friability, grindability, chemical analysis, and high- and low-temperature carbonization assays of Alabama coals have been pointed out.[13] The friability, slacking characteristics, low-temperature carbonization assays, and proximate and ultimate analyses of typical subbituminous coals from 14 mines in the Denver (Colo.) region have been determined.[14] The 14 mines sampled are described briefly.

Classification

The Bureau cooperated with the American Society for Testing Materials and the American Standards Association in developing recently adopted standards for classification of coals.[15]

A revision[16] of Report of Investigations 3296, Classification Chart of Typical Coals of the United States presents the latest (1938) American Society for Testing Materials Standard Specifications for Classification of Coals by Rank (D 388-38; A.S.A. M20.1-1938), with charts showing British thermal units per pound on the moist, mineral-matter-free basis plotted against percentage of fixed carbon on the dry, mineral-matter-free basis. Included also are tables listing the source and rank of 316 typical coals of the United States and the proximate and ultimate analyses and heating values of 51 of these 316 coals not published previously. The higher-rank coals are classified according to fixed carbon on the dry, mineral-matter-free basis, and the lower-rank coals according to British thermal units on the moist, mineral-matter-free basis, that is, containing their natural bed moisture but free of visible surface moisture. Agglomerating and weathering indexes are used to differentiate between certain adjacent groups.

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[13] Hertzog, E. S., Cudworth, J. R., Selvig, W. A., and Ode, W. H., Friability, Grindability, Chemical Analyses, and High- and Low-Temperature Carbonization Assays of Alabama Coals: Bureau of Mines Tech. Paper 611. In press.

[14] Parry, V. F., and Goodman, J. B., Friability, Slacking Characteristics, and Low-Temperature Carbonization Assays of Subbituminous Coals of the Denver (Colo.) Region: Bureau of Mines Rept. of Investigations 3457, 1939, 12 pp.
Parry, V. F., and Goodman, J. B., Subbituminous Coal of the Denver (Colo.) Region. Friability—Slackening (Slacking) Characteristics—Low Temperature Carbonization Assays: Mines Mag., vol. 29, July 1939, pp. 369-374.

[15] American Society for Testing Materials, Report of Sectional Committee on Classification of Coals A.S.A. Project M20: Proc., vol. 38, pt. 1, 1938, pp. 412-413.

[16] Fieldner, A. C., Selvig, W. A., and Frederic, W. H., Classification Chart of Typical Coals of the United States: Bureau of Mines Rept. of Investigations 3296R, 1939, 22 pp.

Application of the Bureau of Mines agglomerating index of coals to coal classification and correlation with fuel-bed observations of the caking action of coals in various types of coal-burning equipment have indicated in a broad sense the practical value of this index.[17]

Origin and Petrographic Composition

Complete columnar sections from top to bottom of the Pittsburgh,[18] High Splint,[19] Sewell,[20] and Pocahontas No. 3 and No. 4 beds[21] were examined macroscopically, and thin sections prepared from these columns were studied microscopically as part of the survey of the gas-, coke-, and by-product-making properties of American coals. Eight other columns from miscellaneous coals were sectioned and examined. These included the Stockton-Lewiston, Monarch, and Pittsburgh beds (Apex mine) and five cannel coals.

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[17] Barkley, J. F., and Burdick, L. R., The Agglomerating Index of Coal: Bureau of Mines Rept. of Investigations 3424, November 1938, 8 pp.; Proc. 24th Fuel Eng. Conf., Appalachian Coals, Inc., Kalamazoo, Mich., December 13, 1938, 8 pp.

[18] Fieldner, A. C., Davis, J. D., Brewer, R. E., Selvig, W. A., Reynolds, D. A., and Sprunk, G. C., Carbonizing Properties and Petrographic Composition of Pittsburgh Bed Coal from Bureau of Mines Experimental Mine, Bruceton, Allegheny County, Pa.: Bureau of Mines Tech. Paper 594, 1939, 43 pp.

[19] Fieldner, A. C., Davis, J. D., Reynolds, D. A., Selvig, W. A., Sprunk, G. C., and Auvil, H. S., Carbonizing Properties and Petrographic Composition of High Splint Bed Coal from Closplint Mine, Closplint, Harlan County, Ky.: Bureau of Mines Tech. Paper 599, 1939, 38 pp.

[20] Fieldner, A. C., Davis, J. D., Selvig, W. A., Brewer, R. E. Holmes, C. R., Reynolds, D. A., and Sprunk, G. C., Carbonizing Properties and Petrographic Composition of Sewell Bed Coal from Wyoming Mine, Wyoming County, W. Va., and the Effect of Blending This Coal with Alma Bed Coal: Bureau of Mines Tech. Paper 601, 1939, 45 pp.

[21] Fieldner, A. C., Davis, J. D., Reynolds, D. A., Schmidt, L. D., Brewer, R. E., Sprunk, G. C., and Holmes, C. R., Carbonizing Properties and Petrographic Composition of Pocahontas No. 3 Bed Coal from Buckeye No. 3 Mine, Wyoming County, W. Va., and of Pocahontas No. 4 Bed Coal from No. 4 Mine, Raleigh County, W. Va.: Bureau of Mines Tech. Paper 604. In press.

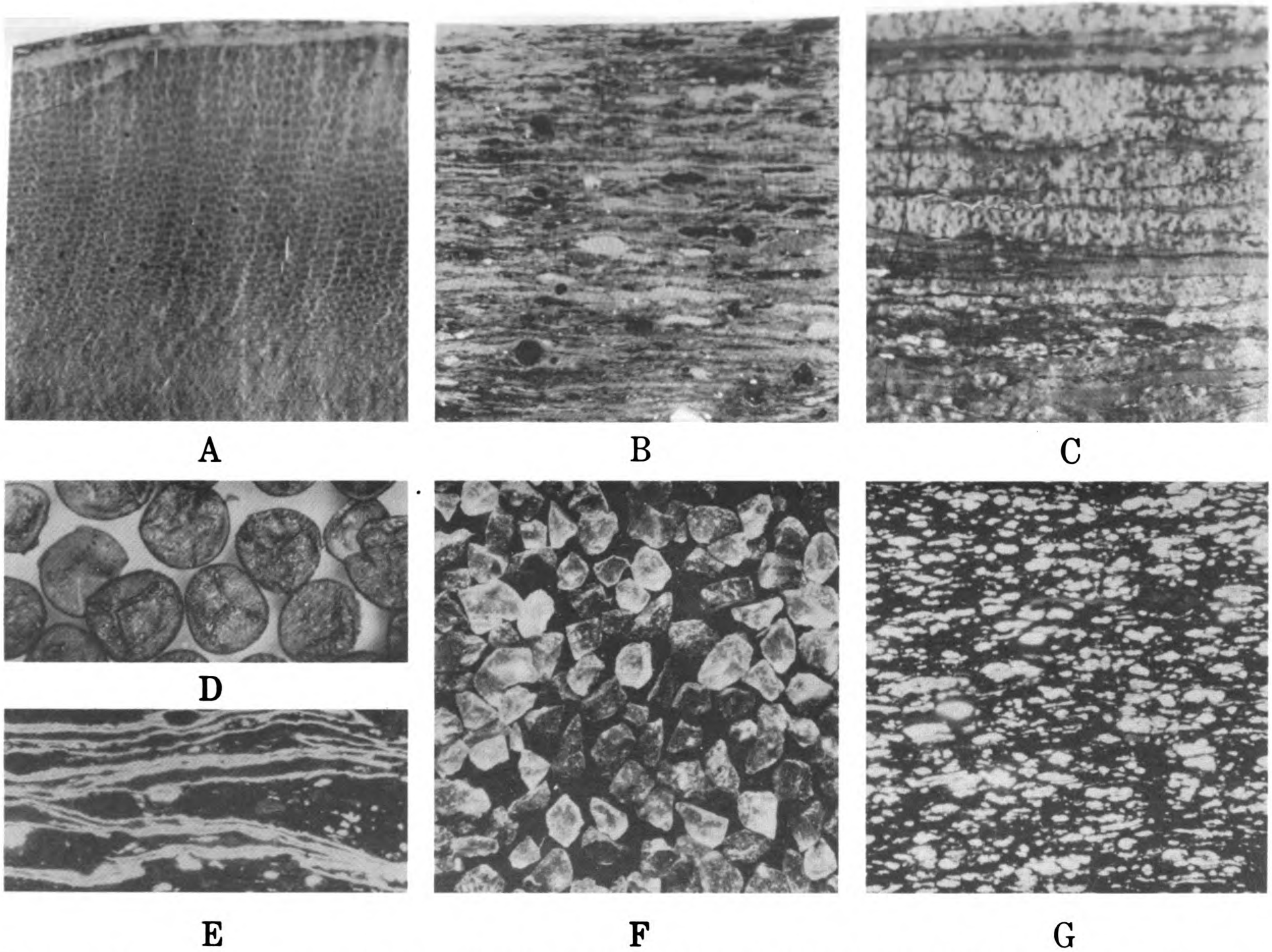

Figure 1.– Coal constituents that are easily liquefied in the small-scale hydrogenation tests: A, Anthraxylon; B, woody degration material; C, leaf residues; D, spores; E, cuticles; F, resins; G, oil algae. (A, B, C, F, and G, 100 X mag., D, 12 X mag., E, 5 X mag.).

A bulletin[22] prepared in cooperation with the Pennsylvania State College describes the results of both noncatalytic and catalytic (2 percent added stannous sulfide) hydrogenation of petrographic constituents of Pittsburgh-bed (Bruceton-mine) coal. The samples ranged from a "pure" anthraxylon to a splint fraction of 30 percent opaque-attritus content. Hydrogenation was carried out in both cases with pressures of the order of 2,000 pounds per square inch and a range of temperature in the vicinity of 450° C. Provision was made for removing light oil and water from the hot hydrogenation residues, thus more nearly approaching the methods employed in continuous-type hydrogenation than does the usual batch-type procedure. This modification resulted in incomplete conversion of the fusain-free organic matter to liquid products in the noncatalytic hydrogenation tests. The material precipitated during hydrogenation showed solubility properties and amounts present that lead to the belief that it originated from the resinous material of the coal. Catalytic hydrogenation, on the other hand, resulted in complete conversion of the fusain-free organic matter in tests on "pure" anthraxylon and improved conversions on other samples. A straight-line relationship was found between the percentage of residue from catalytic hydrogenation and the percentage of opaque attritus in the original sample. The "precipitated" material observed in noncatalytic hydrogenation was almost completely converted by catalytic hydrogenation. Higher hydrogenation temperatures for noncatalytic experiments increased conversion appreciably only for those samples that produced appreciable amounts of precipitated material; only slight increases resulted for catalytic experiments, indicating the extreme resistance of certain portions of the coal hydrogenation. Greater yields of lower-boiling oils and smaller yields of gas resulted from the catalytic than from the noncatalytic treatment. A method for direct determination of water in the hydrogenation products was described; the data indicated that about two-thirds of the oxygen in the coal appeared as water. Hydrogen absorption was greater for the catalytic than for the noncatalytic experiments and occurred in liquid fractions. Petrographic properties of the original samples and of the residues from both catalytic and noncatalytic treatment are described.

Eighty-three samples of pure constituents and types of coal were isolated and studied microscopically in connection with tests of their relative amenability to hydrogenation in the Bureau of Mines laboratory at Pittsburgh, Pa.

Small-scale hydrogenation tests indicated that, with respect to liquefaction yields, the coal constituents may be classed conveniently into two groups. The first group, of easily liquefiable constituents, includes anthraxylon and all the organic portion of translucent attritus. Figure 1 shows the typical appearance of such constituents at various magnifications under the microscope.

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[22] Wright, C. C., and Sprunk, G. C., Hydrogenation of the Petrographic Constituents of Pittsburgh Seam Coal: Pennsylvania State Coll. Min. Ind. Exp. Sta. Bull. 26, 1939, 32 pp.

The second group of constituents is more difficult to liquefy and includes opaque attritus and fusain. Opaque attritus forms the ground mass of splint coals. It is not a homogeneous material. In medium-thin sections it is opaque, and in extremely thin sections a portion of it is semitranslucent and brown. Other fragments can be found in various degrees of translucency; and some, even in the thinnest sections, appear to rival fusain in opacity. Figure 2 illustrates the physical appearance of these constituents at the magnifications indicated.

Small-scale hydrogenation tests show that American splint coals are less suitable for hydrogenation than bright coals, and that the opaque attritus is the principal constituent, other than fusain, responsible for the low liquefaction yields obtained with some coals of low and intermediate bituminous rank. Considerable range in the "degree of opacity" explains the variation in yield from different splint coals. All of the hydrogenation residues from splint coals have a similar appearance under the microscope. The individual particles usually are less than 5 microns in diameter, although they may be grouped in larger masses. Figure 3 shows that they are smaller and more rounded than the sharply angular particles of hydrogenation residue from fusain.

More than 450 thin sections of western low-rank coals were prepared. All of the coals are more or less resinous, many have less than 1 percent fusain, and a number are exceptionally low in opaque attritus.

Further correlations between coal petrography and coal hydrogenation are described in the section "Hydrogenation of Coal".

Resins from a British Columbia and a Utah coal were separated by flotation and analyzed. Fischer low-temperature assays were made for yields of carbonization products. The carbonized residues are low, as would be expected from the low fixed-carbon content of the resins. The gas from the British Columbia resin contains more carbon dioxide and carbon monoxide than that from the Utah resin, probably owing to its higher oxygen content. The yields of tar for the British Columbia and Utah resins are 194 and 178 gallons per ton (dry basis), respectively, compared with 86 gallons per ton (dry basis) from pure spores separated from a Michigan coal

Fischer low-temperature carbonization assays and agglutinating-value tests were made on layer samples isolated from columnar sections of Pocahontas No. 3 and Stockton-Lewiston coal beds of West Virginia and of Lower Banner-bed coal of Virginia. The splint-coal layers from the West Virginia coals yield considerably less gas on carbonization, and their agglutinating values are lower than those of the bright-coal layers from these coals. The differences are more pronounced than those observed in tests on coals from other beds. Petrographic examination of the column sample from Lower Banner bed of Virginia shows that considerable woody cannel coal is present. The cannel coal gives a lower agglutinating value and less swelling in the Fischer assay than the rest of the column sample; the yields of carbonization products are similar.

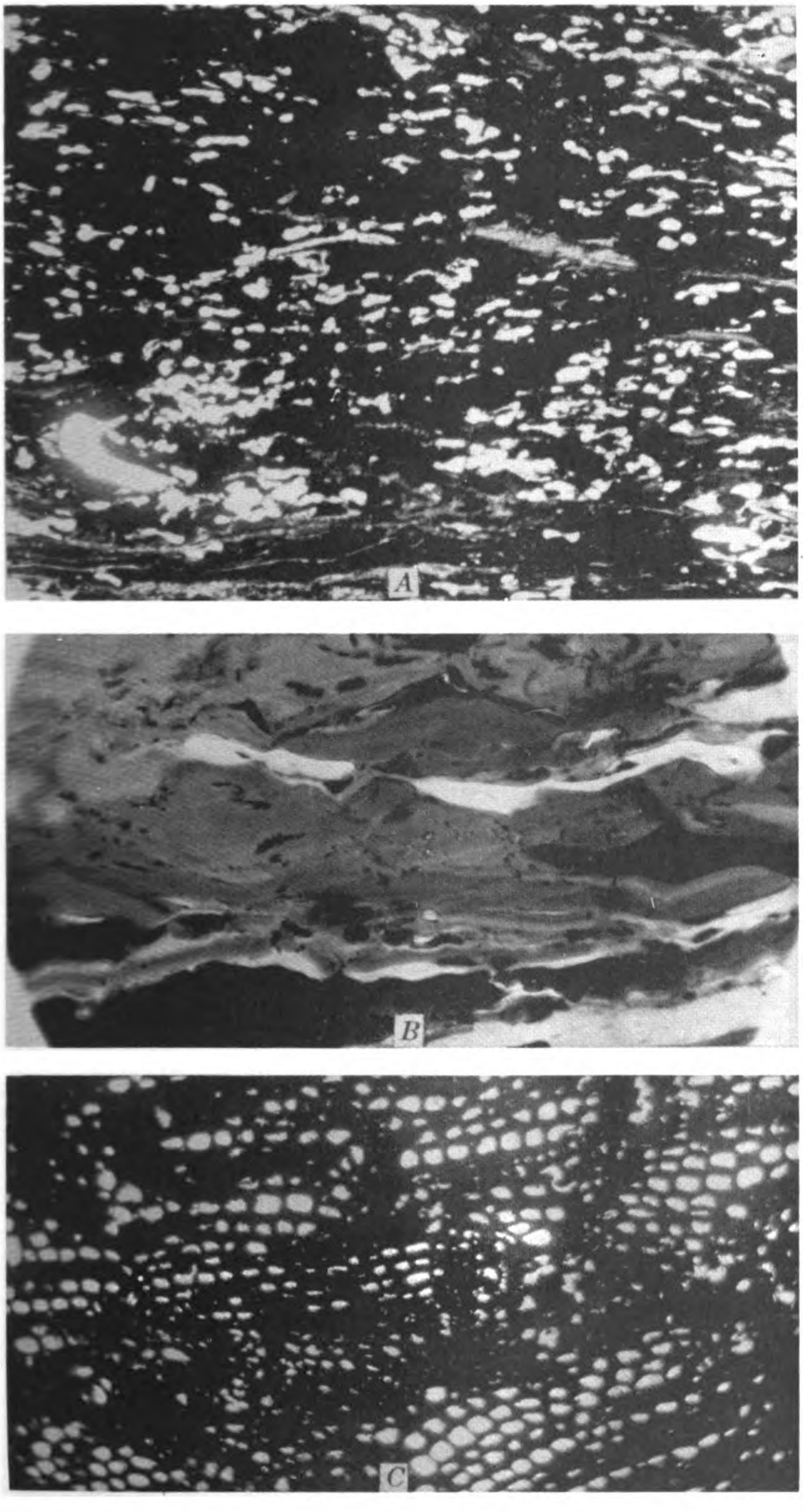

Figure 2.- Coal constituents that are difficult to liquefy in small-scale hydrogenation tests: A, Opaque attritus (200 X mag.); B, brown matter in exceedingly thin section (1,000 X mag.); C, fusain (150 X mag.).

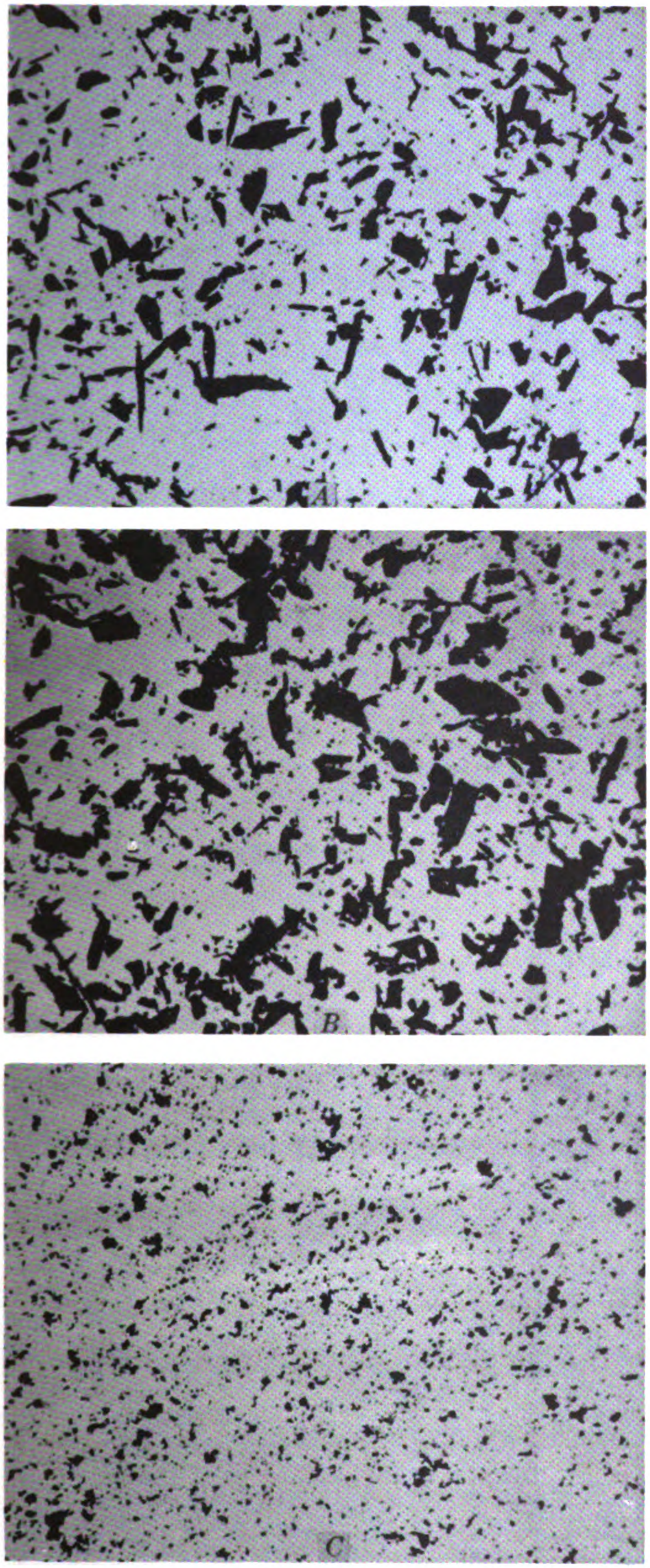

Figure 3.- Fusain and hydrogenation residues from fusain and splint coal (150 X mag.): A, Hand-picked fusain; B, hydrogenation residue from fusain; C, hydrogenation residue from splint coal.

The results of crucible swelling tests and the degree of expansion in experimental ovens of the sole-heated and slot type were compared for seven bituminous coals of widely varying rank. Figure 4 shows the carbonized residues of the crucible tests carried out according to the methods there indicated. The order of increasing rank of the coals is G, E, B, F, C, D, and A, which is approximately the same order as their relative order of increasing expansion. The crucible tests show that coals G, E, and A in general swell less than coals B, F, C, and D. The expansion tests indicated that coals G and E were contracting and that coal A gave the highest expansion of any of the seven coals. The comparative results of the two types of tests indicate that there is considerable difference between the swelling of coal in the crucible tests, where all the coal becomes plastic at nearly the same time and is allowed to expand freely, and the expansion of coal in a somewhat restricted space similar to that of a coke oven, where the coking is progressive and the zone of plastic material is moving through the coal charge.

Application of the British Standards Institution crucible swelling test to determine the relative caking properties of coal when burned on grates gave promise of usefulness. Two coals of similar chemical composition from the Pocahontas bed showed swelling numbers of 4 and 9+, respectively. The first gave satisfactory results when burned; the second gave trouble, owing to caking on the grates.

Plasticity

The plastic properties of all coals studied in the survey of the carbonizing properties of American coals during the year were determined by the revised Agde-Damm dilatometer, Davis plastometer, and Gieseler test methods.[23] In addition, many tests by these methods were made in connection with special studies of oxidized coals, of bright and splint layer samples, and of medium- and low-volatile expanding coals, including screened portions of certain coals of low-volatile rank. Seven coal samples also were tested in cooperation with Subcommittee XV of the American Society for Testing Materials Committee D-5 in connection with standardizations of apparatus and procedure.

With increasing extent of oxidation, the plastic properties of bituminous coking coals were affected as follows: (1) Increase in both the initial softening and fusion temperatures and a small decrease in the temperature marking the end of the plastic temperature range; (2) decreasing differences between initial softening and fusion temperatures from series to series with increasing rank of the coals; (3) smaller reductions in linear expansion (Agde-Damm method) for high-volatile A than for medium-volatile coals; and (4) reduction in fluidity of all coals - most pronounced

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[23] Brewer, R. E., and Triff, J. E., Measurement of Plastic Properties of Bituminous Coals. Comparison of Gieseler and Davis Plastometer and Agde-Damm Dilatometer Methods: Ind. and Eng. Chem., Anal. Ed., vol. 11, May 15, 1939, pp. 242-247.

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for coals of highest rank. These changes in plastic properties correspond to changes in coking power of the coals. The coking power of high- and medium-volatile coals is improved by a limited amount of oxidation, whereas with low-volatile coals slight oxidation decreases the coking power.

Plastic properties were determined on three high-volatile A, one medium-volatile, and two low-volatile bituminous coals, and also on bright and splint layer samples separated from the columnar sections of these six coals. Three 100-percent splint layer samples showed no fusion. Three other 100-percent splint layers, all six of the composite samples, and seven predominantly bright layer samples showed well-defined plastic properties. Classified according to their content of anthraxylon and their anthraxylon to attritus ratios, it was observed that: (1) The nonfusing group all contained 6 area percent or less of anthraxylon and had an anthraxylon to attritus ratio of 0.07 to 1 or lower; and (2) the fusing group all contained 9 area percent or more of anthraxylon and had an anthraxylon to attritus ratio 0.10 to 1 or higher. The relationship between the contents of anthraxylon and the ratios of anthraxylon to attritus is fairly regular, and with increase in these values the percentage of splint coal drops. The relative order of fusion temperatures for the samples of a given series - composite coal, bright coal, and splint - varies with the predominating type of coal present. In some series the fusion temperatures are lowest for the composite coal; in others they are lowest for the samples containing the highest percentage of bright coal. The order of decrease in expansion (Agde-Damm test) was from bright to composite to splint coal.

Some difficulty has been experienced in obtaining close checks by the Davis plastometer test method on low-volatile bituminous coals of high fusain content. It was believed that the higher specific gravity of fusain and its relatively fine state of subdivision - resulting from ease of grinding this friable material - as compared with the rest of the coal sample, might permit segregation of the fusain during testing of such coals in the Davis plastometer rotating retort. To test this hypothesis, the original through-20-mesh sample was screened into two portions: (1) Through 20 and on 35 mesh and (2) through 35 mesh. The plastic properties of these screened portions were determined by the Davis plastometer, and the results were compared with those for the original 20-mesh sample, and also for this sample ground to pass through a 35-mesh screen. The through-35-mesh material screened from the original 20-mesh sample gave a higher fusion temperature and a lower maximum resistance than the other three samples. This result supports the argument that fusain was more concentrated in the finer sizes of the original sample; and, because of its higher specific gravity, the fusain segregated during the determination of the plastic properties of the sample in the Davis plastometer rotating retort.

COAL MINING

During the fiscal year study of many problems relating to safe and economical practices in the mining of coal has continued. A greater

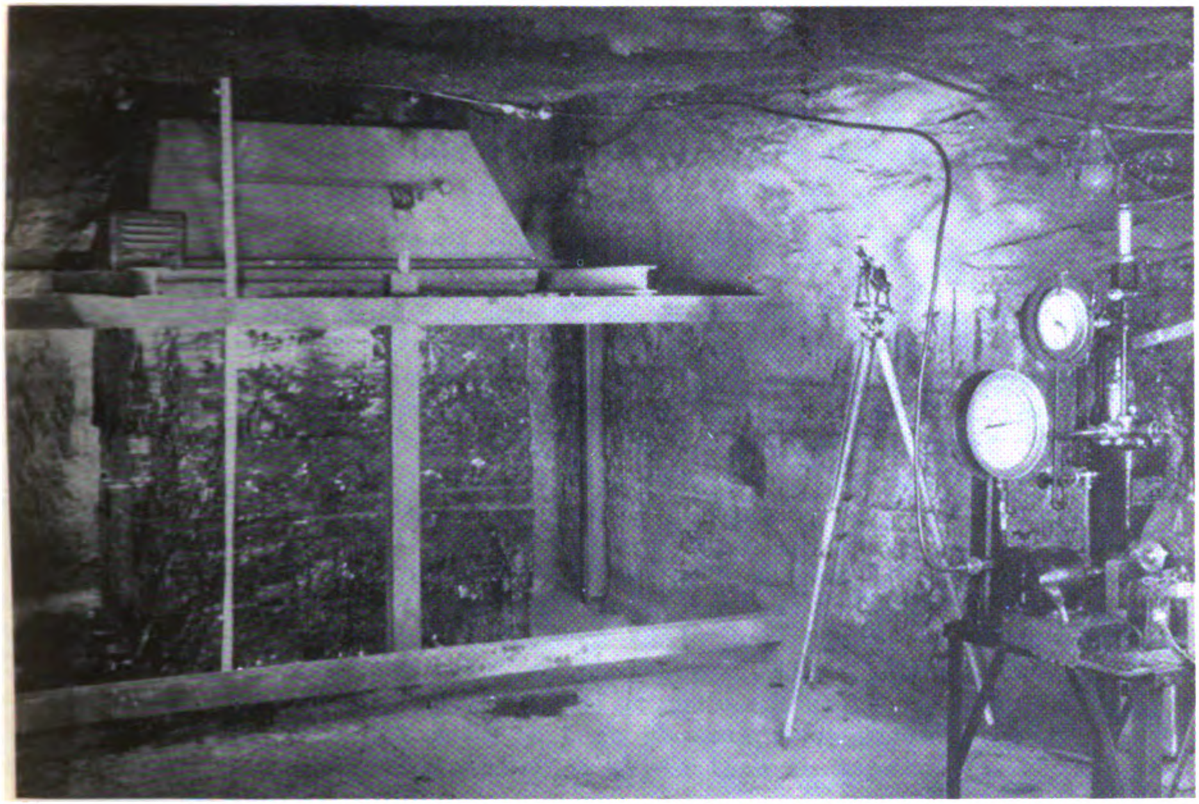

Figure 5.- General view of pillar and loading assembly during test.

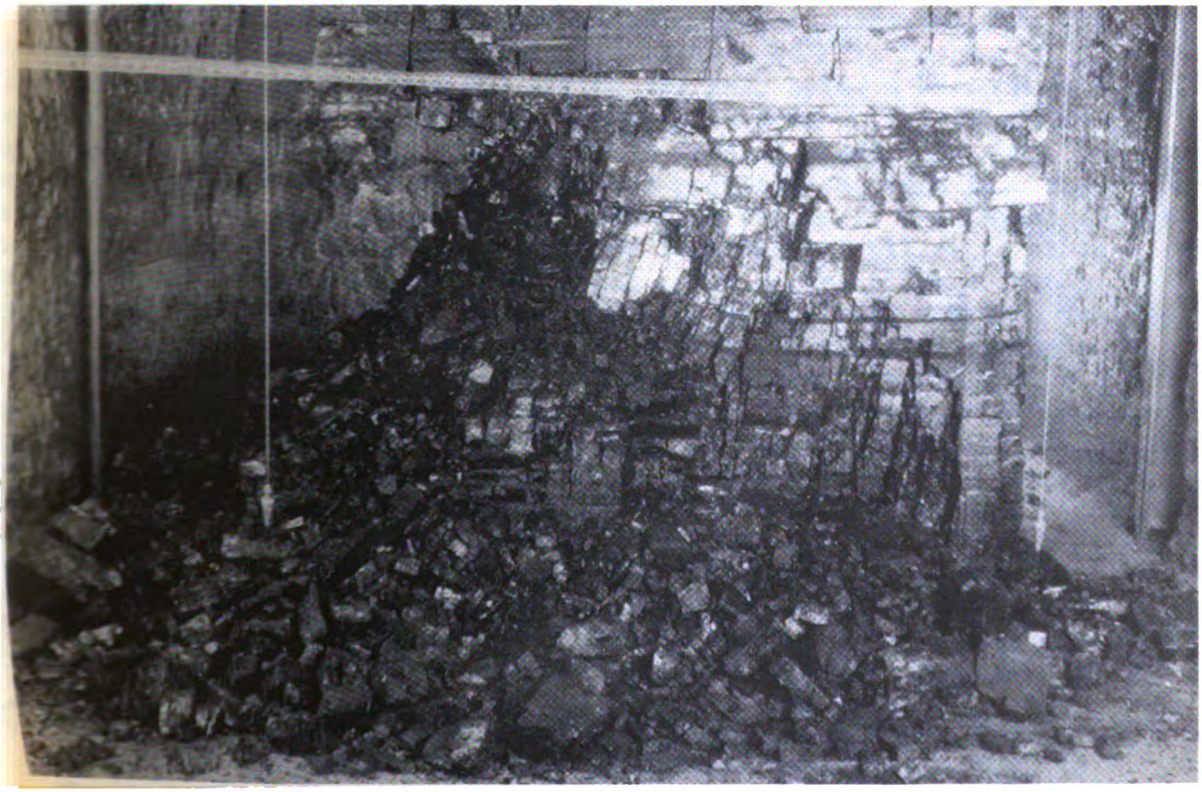

Figure 6.- Close view of base of pillar after failure.

amount of cooperative work between the Bureau of Mines and a number of States and mining companies has resulted in a well-balanced, progressive program.

Experimental Coal Mine

During the fiscal year the Experimental Coal Mine Section cooperated in furnishing coal to the Hydrogenation Section, in testing lighting equipment and gas-detecting devices with the Electrical-Mechanical Section, and in the taking of motion pictures underground by the Safety Division of the Bureau. Four large-scale demonstrations of the explosibility of coal dust were held before approximately 1,000 visitors, and 200 other visitors were received at the Experimental mine during the year.

Three major research investigations were conducted during the year in the Experimental coal mine on problems relating to safety in mining and other industries. These were (1) large-scale determinations of the strength of pillars of the Pittsburgh coal bed in place; (2) both large- and small-scale determinations of the inflammability and explosibility of dusts; and (3) large-scale determinations of certain phenomena accompanying gas explosions such as may initiate coal-dust explosions in mines.

Bearing-Strength and Compressibility Tests in the Mine

The early stages of this investigation have been reported.[24] The work was continued on successively larger pillars of Pittsburgh-bed (Bruceton-mine) coal, the last one during the year being a 64-inch cube equal in height to that of the bed. Figure 5 is a view of this pillar during the early part of the test. A total load of 2,835,000 pounds or unit load of 695 pounds per square inch was required to crush it. Figure 6 is a close view of the base of the pillar after failure. The investigation showed that the ratio of lateral dimension to height of pillar is an important factor in determining its strength. For square pillars resting on a solid floor and comprising the entire height of the coal bed, the following relation was found: $S = K\sqrt{R}$, in which S is the crushing strength of this coal in pounds per square inch, K a constant equal to 695, and R the ratio of lateral dimension to height of pillar. Other tests indicated that the value of K may change with the thickness of the bed, becoming larger as the bed becomes thinner. The unit strength of the 64-inch cube was only one-fourth to one-third of that shown in laboratory tests of small cubes of coal made by a number of investigators. The present data indicate that a pillar the height of the bed (64 inches) in the Experimental mine would have to be 45 feet square before its unit strength reached 2,100 pounds per square inch. It was found that continued lateral expansion of a pillar of coal under constant load is a much better indication of approaching failure than vertical compression. A detailed description of this invest gation has been issued.[25]

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[24] Fieldner, A. C., Annual Report of Research and Technologic Work on Coal, Fiscal Year 1938: Bureau of Mines Inf. Circ. 7052, 1939, 44 pp.

Inflammability and Explosibility of Pitch Dust

An extensive investigation of the inflammability and explosibility c dusts prepared from pitches produced from coal tar, water-gas tar, and pe troleum has been conducted during the year. The basis of comparison was Pittsburgh coal dust. The pitch dusts and the coal dust were ground to comparable sizes before being tested.

The lower quantity limit of inflammability of a sample of coal-tar pitch and one of water-gas-tar pitch tested on a large scale in the Exper mental coal mine lay between 0.040 and 0.053 ounce per cubic foot, wherea that of Pittsburgh coal dust lies between 0.053 and 0.067 ounce. Both pitches attained maximum explosibility with a concentration of 0.093 ounc compared with 0.120 ounce for Pittsburgh coal dust. These latter quantit are close to the amounts required to consume all the oxygen from the air. With concentrations giving maximum explosibility, 80 percent incombustib was required in mixtures of the pitch dusts to prevent propagation of flame compared with 77 percent for Pittsburgh coal dust. The pitch dust are slightly more inflammable than the coal dust, but the precautions taken to prevent explosions when pulverized coal is fired are applicable to and adequate for the pitch dusts.

Laboratory tests were made on four other samples of coal-tar pitch dust. The inflammability of the dusts had approximately a straight-line relation to their volatile content as determined by proximate analysis. Tests were made also with atmospheres of reduced oxygen content. Pitch dusts failed to ignite with 18.4 to 13.3 percent oxygen; Pittsburgh coal failed with 16.8 percent oxygen. Petroleum-pitch dust was found to be more inflammable than the coal-tar-pitch dusts.

Many other laboratory tests were made. A detailed report of the in vestigation will be published.

In addition to the work on pitch dust, laboratory determinations of the inflammability of dusts from such materials as petroleum coke, charcoal, metallic salts of tartaric and acetic acids, and transformer vault were continued.

Gas Explosions

Complete data were obtained for explosions of uniform mixtures of natural gas and air confined by a paper diaphragm in the Experimental mi The enclosed volume was approximately 1,300 cubic feet. The percentage gas ranged from the lower to the upper limit of inflammability in different tests; ignition was at the face in all cases. Greatest length of flame was 170 feet, seven times the length of the gas zone, and occurred with 10 and 11 percent gas in the gas-air mixtures. Greatest pressure was recorded at the point of ignition; it was only 2 pounds with 7 percent gas, rose to a maximum of 18 pounds with 11 percent gas, and fell rapidly to 2 pounds with 14 percent gas.

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[25] Greenwald, H. P., Howarth, H. C., and Hartmann, I., Experiments on the Strength of Small Pillars of Coal in the Pittsburgh Bed: Bureau of Mines Tech. Paper 605, 1939, 22 pp.

Explosibility of Coal Dust

Comparison[26] of the American and British large-scale test methods for determining the explosibility (inflammability) of coal dust and the effect of various factors pertinent to the results obtained by these test methods shows that the principal factors are the size and composition of the coal dust, the quantity of coal dust present, the effect of inflammable gas in the air current, the effect of varying strength of the source of ignition, the manner of distribution of dust in the test gallery or passageway, and the effect of changes in the cross section and configuration of the gallery or passageway. The last three factors have not been investigated fully. The proportion of incombustible material that must be mixed with coal to prevent propagation of flame depends on particular test conditions. Laboratory test data should include a reasonable factor of safety when applied to commercial coal-mining operations.

Rock Dusting

Rock dusting back entries of coal mines against coal-dust explosions by various hand, high-pressure machine, and low-cost (labor-saving and semi-automatic or automatic) methods was discussed with particular reference to effectiveness and cost.[27]

Movement of Roof

Earlier studies in a mechanized mine were reported last year.[28] The second mine mentioned in that report was the Crucible mine of the Crucible Fuel Co. In this mine all coal is loaded by hand, with consequent slower movement of rib lines. Because of this and the different manner in which pillars are split, roof movement in this mine is more irregular, but the basic principles are the same as those in a mechanized mine. Convergence of roof and floor during pillar extraction was measured in three sections of the Crucible mine.[29] In one newly opened section considerable trouble was experienced with heaving floor. The cause could not be determined readily. It was suggested that it might be the effect of stress residual in the coal bed from past geological disturbances or that the fireclay floor may have been overloaded from the heavier cover in this section compared with other parts of the mine. Extensive measurements of surface subsidence were made over the underground areas studied. With a cover of 300 to 500 feet, subsidence lagged behind extraction of the coal; the surface sank over the goaf in a long, sweeping curve. The surface movement was 40 to 50 percent of the thickness of coal removed beneath. The data indicate that a large amount of the weight of the strata over the goaf is carried by coal pillars along the rib line. The cover contains at least two thick and strong rock members - the Pittsburgh sandstone and the Benwood limestone. Subsidence of the surface can therefore be minimized by leaving strong pillars behind the rib line as long as is consistent with their removal in an economical manner when the rib line reaches them.

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[26] Greenwald, H. P., Notes on Large-Scale Tests of the Explosibility of Coal Dusts Made in the United States and Great Britain: Bureau of Mines Rept. of Investigations 3462, 1939, 9 pp.

[27] Greenwald, H. P., Discussion of "What Are the Most Efficient Methods of Rock-Dusting Back Entries" by J. V. McKenna: Proc. Coal Min. Inst. America, 1938, pp. 67-70.

[28] Fieldner, A. C., work cited in footnote 24, p. 15.

[29] Maize, E. R., and Greenwald, H. P., Studies of Roof Movement in Coal Mines, 2. Crucible Mine of the Crucible Fue Co.: Bureau of Mines Rept. of Investigations 3452, 1939, 19 pp.

Fundamental information obtained from studies of ground movement and subsidence in coal mines has been found useful in interpreting the recent violent outbursts with "heaving shales" in the East Texas oil field.[30]

American Institute of Mining and Metallurgical Engineers Technical Paper 1014, Ground Movement and Subsidence Studies in Mining Coal, Ores, and Nonmetallic Minerals, originally published in February 1937, has been reprinted.[31]

Methods

A comprehensive study based on the personal observation of the Burea former Chief Mining engineer of practices in different coal fields of Eur and under various economic and regulatory conditions of mining compared w those in the United States was completed.[32]

The advantages and disadvantages of the various types of equipment a mining systems now used at 11 bituminous-coal mines in the United States have been analyzed in relation to the character of certain physical conditions peculiar to the different mines.[33] The ultimate success of mecha zation depends on thorough understanding of the problems invilved, coord nation of all operations - cutting, shooting, timbering, loading, and tr portation - and proper training of men to operate efficiently and safely the various machines used.

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[30] Rice, G. S., Ground Movement and Subsidence. (Old Mining Problem Spreads to East Texas Oil Field): Min. and Met., vol. 20, January 1 p. 8.

[31] Rice, G. S., Ground Movement and Subsidence Studies in Mining Coal, and Nonmetallic Minerals: Min. Technol., vol. 3, January 1939, 15 p

[32] Rice, George S., and Hartmann, Irving, Coal Mining in Europe: Burea of Mines Bull. 414, 1939, 369 pp.

[33] Toenges, A. L., and Jones, F. A., Multiple-Shift Mechanical Mining i Some Bituminous Coal Mines. Progress Rept. 2: Bureau of Mines Inf. Circ. 7067, 1939, 58 pp.

The work that has been done in certain States, notably Kansas, Missouri, Indiana, and Illinois, in reclaiming the surface of land from which coal has been removed by strip mining was described.[34] Natural restoration and planned reclamation methods are reviewed, which have reclaimed many acres for grazing, recreation, forest reserves, and, in some places, agriculture. The latter two phases already have produced "money crops".

Fires in Anthracite Mines

The investigation of the causes, behavior, and control of fires in anthracite mines was continued. Data on several additional fires were obtained, tentative plans for attacking a large, active fire with inert gas were formulated, and one fire area was inspected before being opened.

Data obtained in previous low-temperature oxidation tests were used in calculating the heat of oxidation at temperatures between 100° and 450° C. by two different but related methods.[35] The calculations were based upon the amount of oxygen consumed and the heating value of the coal before and after oxidation. Test data included 25 anthracites and 8 lower-rank coals. After about 1,600 cubic feet of oxygen was consumed per ton of coal the heat of oxidation was virtually constant at about 4.1 calories per cubic centimeter (NTP) of oxygen consumed.

Observations that many anthracite refuse banks, some very high and large in area, and a few more than 50 years old, have never caught fire and that laboratory ignition temperatures of anthracite and anthracite refuse products are high - usually higher than 400° C. - indicate that spontaneous ignition of such materials is improbable. The lack of evidence that anthracitic materials will heat spontaneously to the temperature of ignition suggested a study of other causes of fires in anthracite refuse banks.[36] External sources of heat and spontaneous combustion of materials having low-ignition temperatures are considered. Laboratory determinations of the ignition temperatures of bituminous and subbituminous coals and of various cellulosic materials, such as paper, wood, discarded mine timber, sawdust, oil, grease, and brattice cloth, show that these materials ignite at much lower temperatures than do the principal materials in anthracite refuse bank. Such materials in the bank are fire hazards. Recommendations are made for decreasing the possibility of fire by proper methods of storage and suitable control tests.

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[34] Toenges, A. L., Reclamation of Stripped Coal Land: Bureau of Mines Rept. of Investigations 3440, 1939, 11 pp.

[35] Scott, G. S., Heat Liberated in the Low-Temperature Oxidation of Anthracite: Bureau of Mines Inf. Circ. 7053, 1939, 10 pp.

[36] Jones, G. W., and Scott, G. S., Chemical Considerations Relating to Fires in Anthracite Refuse: Bureau of Mines Rept. of Investigations 3468, 1939, 13 pp.

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The absence of carbon monoxide in the atmosphere of sealed fire areas is not absolute evidence that the fire has been extinguished.[37] Tests are described showing that certain types of bacteria or micro-organisms, present in anthracite gangway waters, sewage, and surface materials can oxidize carbon monoxide to carbon dioxide and hydrogen to water. Under favorable conditions, this disappearance of carbon monoxide and hydrogen in sealed fire areas may be as rapid as the generation of carbon monoxide by the fire.

The bacteria that cause oxidation of carbon monoxide and hydrogen have been isolated and studied microscopically.[38] A study of the sources of 43 samples, including manhole sludges, mine waters, sewage, soil, tap and distilled waters, and tests upon these to determine their ability to oxidize hydrogen and carbon monoxide indicate that the activity of the samples was due to certain soil bacteria contained in surface drainage contaminations. The samples were tested first in small sterilized bottles containing an atmosphere of carbon monoxide, hydrogen, and air, then returned to their original containers and stored in the dark for 2 to 6 months, and finally retested similarly in 5-gallon bottles, in many instances in the presence of sponge to increase the surface. In both the small- and large-bottle experiments the final gas was lower in hydrogen, carbon monoxide, and oxygen and higher in carbon dioxide. The increase in nitrogen percentage corresponded in general to the net decrease in volume caused by the reactions. All of the large-bottle experiments in which sponges were added showed an increased rate of elimination of hydrogen and carbon monoxide. In some of the large-bottle tests the activity was much less than in the earlier corresponding tests of the same sludges in the small-bottle experiments. It was believed that this was due to depletion of the necessary mineral matter or that higher forms of life consumed the bacteria. Certain scums were observed to contain rotifers, nematodes, and protozoa. These motile, higher forms of life appeared to be consuming the carbon monoxide and hydrogen bacteria. After their elimination, the bacteria again grew and multiplied in the presence of carbon monoxide and air.

Figure 7 is a photomicrograph (X 1,000) showing the bacteria in a scum formed on the surface of a manhole sludge. They appear as short, almost spherical rods about 0.5 micron in diameter and 1 to 4 microns long, without motion. They seem to be identical with the Bacillus oligocarbophillus described by Beijerinck and van Delden.[39] Since surface drainage con-

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[37] Jones, G. W., and Scott, G. S., Carbon Monoxide in Underground Atmospheres. The Role of Bacteria in Its Elimination: Ind. and Eng. Chem., vol. 31, June 1939, pp. 775-778.

[38] Jones, G. W., and Scott, G. S., Oxidation of Carbon Monoxide and Hydrogen by Bacteria: Bureau of Mines Rept. of Investigations 3466, 1939, 10 pp.

[39] Beijerinck, M. W., and van Delden, A., Concerning a Colorless Bacillus Whose Carbon Nourishment Comes from Atmospheric Air: Centralblatt Bakt., vol. 10, part 2, 1903, pp. 33-47.

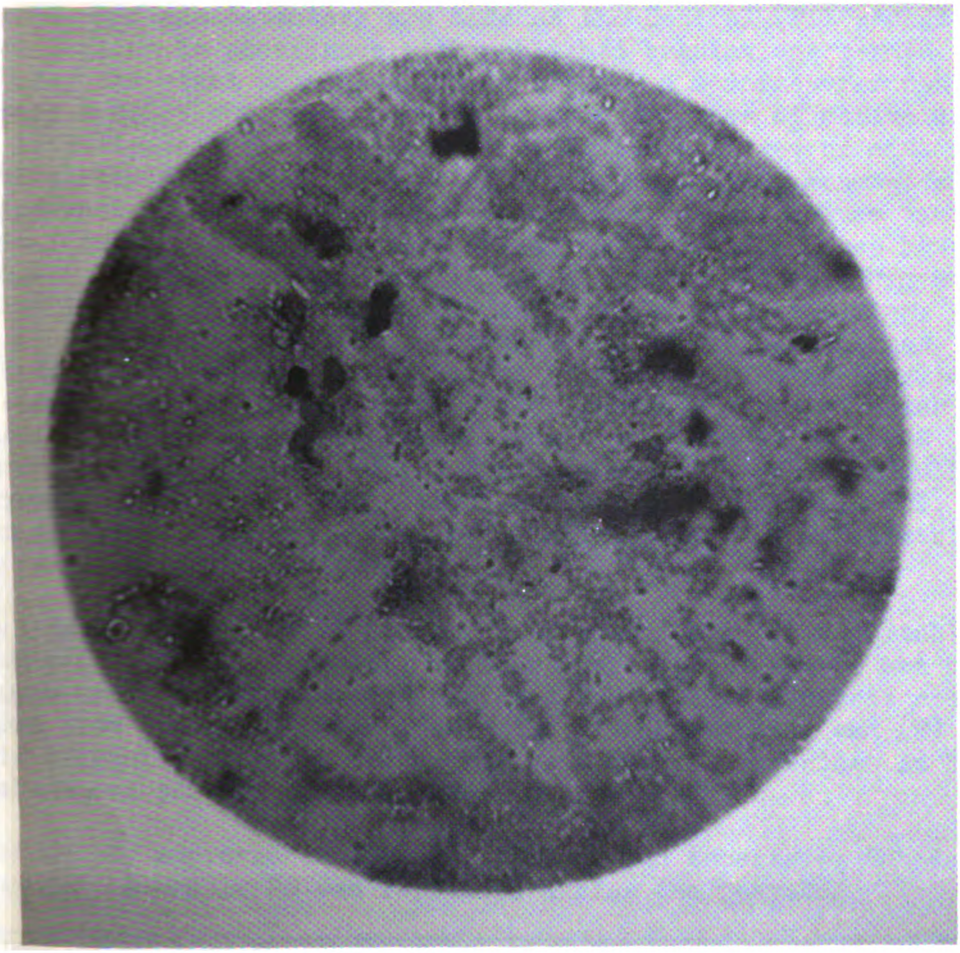

Figure 7.- Photomicrograph of bacteria causing oxidation of carbon monoxide, 1,000 X mag.

taining carbon monoxide-consuming bacteria may get into mine workings, this fact must be taken into account before it may be concluded that on the basis of the absence of carbon monoxide in the gas analysis from a mine-fire area, an underground fire is inactive and it is safe to reopen the mine.

Permissible Mine Equipment

Investigations for safety of electrically operated mine equipment, safety lamps, and gas detectors are made at the Central (Pittsburgh, Pa.) Experiment Station in accordance with published regulations termed "schedules". Under the eight schedules now in effect, a manufacturer can have nearly every type of equipment used at or near the face of active mine workings investigated for its liability to ignite mine gas or coal dust, or a combination of these. The type of equipment tested, general and specific requirements of construction and performance, and procedure of testing leading to final approval (or rejection) by the Bureau of Mines have been described.[40] Forty approvals were issued during the fiscal year under the eight schedules now active, three more than in the preceding year. These included 17 coal-cutting machines, 4 conveyors, 5 loading machines, 1 portable air compressor, 2 pumps, 1 drill, and 1 rock-dust distributor, all completely electrically operated, motor-driven machines approved under Schedule 2D; 2 electric cap lamps under Schedule 6C; 1 electric lantern and 1 flashlight under Schedule 10C; 1 portable "methane tester" under Schedule 8C; and 4 dry-cell-type, single-shot blasting units under Schedule 12B. One extension of approval to include an all-aluminum model of permissible safety lamp was issued under Schedule 7C.

New types of motor-driven equipment approved were (1) a portable, self-contained conveyor (traveling on four rubber-tired wheels and powered by a storage-battery "shuttle car" designed for use in trackless rooms to convey coal from the loading machine to the discharge point and (2) a pump to furnish hydraulic pressure for expanding a special cartridge in breaking down coal without the use of explosives.

Schedule 9B, approved October 25, 1938, which supersedes Schedule 9A, was issued.[41] Schedule 2D was reprinted during the year.[42]

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[40] Ilsley, L. C., Investigation of Electrical Equipment, Safety Lamps, and Gas Detectors for Safety: Bureau of Mines Inf. Circ. 7087, 1939, 15 pp.

[41] Bureau of Mines, Permissible Telephone and Signaling devices: Sched. 9B, approved October 25, 1938, issued February 4, 1939, 6 pp. (Prepared by Electrical-Mechanical Section.)

[42] Bureau of Mines, Explosion-Proof Mine Equipment. Requirements for Approval of Storage-Battery Locomotives and Power Trucks, Junction Boxes, and Electric Motor-Driven Equipment: Sched. 2D, approved May 23, 1936, reprinted April 29, 1939, 29 pp. (Prepared by Electrical-Mechanical Section.)

Numerous changes in design of explosion-proof compartments for incorporation in permissible mining machinery were reviewed and authorized. "Letters of suitability" were issued during the fiscal year for nine motors, two controllers, and one resistor box.

In all, 1,354 explosion tests were made during the fiscal year in the investigations of complete machinery and individual parts.

Engineers of the Electrical-Mechanical Section inspected equipment for which approval had been requested at 16 factories and mines and cooperated with company engineers on problems of developing satisfactory designs for permissible equipment. Field studies of electrical and mechanical hazards associated with the operation of machinery in mines were made at 25 mines and factories.

The advantages of permissible electrical mine equipment in coal mines were pointed out.[43] Such equipment embodies rugged construction features and is adequately and safely wired. Continuity in service and safety in operation have been increased greatly through the extended use of permissible electrical mine equipment.

During the year more than 700 persons, including students, mining officials, and representatives of manufacturers, visited the electrical laboratory, where the approval system was explained and demonstrations were made to show how electric sparks and flashes can ignite methane-air mixtures in a coal mine. Studies were completed of methods of operation of rolling contacts for reducing nipping hazards on self-propelled minin machinery[44] of the conditions to be met in installing various types of high-tension power circuits in coal mines, including recent developments in types of cables;[45] and of the manufacture, inspection, and care of hoisting ropes for mine shafts and slopes.[46] A testing machine was installed to test the strength of ropes after service in mines.

An abstract of the laws and regulations of 29 States on allowable speed when men are hoisted or lowered in mines and the number of men permitted on a cage at one time was published.[47] Distinctive mechanica and electrical signal systems are now available, which avoid the hazard of movement of the cage in response to wrong signals.[48]

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[43] Ilsley, L. C., The Advantages of Permissible Electrical Equipment: Mechanization, vol. 3, No. 6, June 1939, pp. 65-68.

[44] Gleim, E. J., Reducing Nipping Hazards when Tramming Coal-Mining Machinery: Bureau of Mines Inf. Circ. 7056, 1939, 3 pp.

[45] Gleim, E. J., Installation of High-Tension Power Circuits in Coal Mines: Bureau of Mines Inf. Circ. 7044, 1939, 14 pp.

[46] Ilsley, L. C., and Mosier, McHenry, Inspection and Maintenance of Mine Hoisting Ropes: Bureau of Mines Tech. Paper 602, 27 pp.

[47] Ilsley, L. C., and Gleim, E. J., State Regulations Pertaining to Hoisting of Men: Bureau of Mines Inf. Circ. 7057, 1939, 13 pp.

Permissible Explosives and Blasting Devices

At present permissible explosives represent 35 percent of all explosives used in coal mining, an increase of 31 percent over 30 years ago.[49] [50]

The properties of nonpermissible pellet-powder explosive and the hazards involved in its transportation and use, together with the comparative amounts of pellet powder, black blasting powder, and permissible explosives used in the production of coal in West Virginia, Pennsylvania (bituminous and anthracite mines), Ohio, Maryland, and the United States as a whole from 1934 through the first 10 months of 1938, inclusive, have been described.[51]

Schedule 1B (Procedure for Testing Explosives used in Metal Mines, Tunnels, Quarries, and other Engineering Operations, with Test Requirements from Schedule of Fees), Schedule 17C (Procedure for Testing Explosives for Permissibility in Coal Mines, with Test Requirements, Tolerance Limits, and the Schedule of Fees), and Schedule 20 (Permissible Blasting Devices; Procedure in Testing, Fees, and Requirements for Approval) have been incorporated into one schedule, 1C.[52]

PREPARATION OF COAL

Washing in Alabama

The Southern (Tuscaloosa, Ala.) Experiment Station, in cooperation with the University of Alabama and Alabama coal producers, has continued studies on the washability characteristics of important Alabama coals and increasing the yield of marketable coal by re-treating table middlings.

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[48] Mosier, McHenry, and Gleim, E. J., Some Signaling Systems in Metal-Mine Shafts: Bureau of Mines Inf. Circ. 7083, 1939, 5 pp.

[49] Tiffany, J. E., Coal-Mine Explosives: Their Characteristics, Selection, and Safe Use: Proc. Coal Min. Inst. America, 1937, pp. 35-56; Bureau of Mines Inf. Circ. 7040, 1938, 20 pp.

[50] Bureau of Mines Active List of Permissible Explosives and Blasting Devices Approved Prior to June 30, 1938: Rept. of Investigations 3421, 1938, 25 pp. (Compiled by J. E. Tiffany and Z. C. Gaugler.)

[51] Tiffany, J. E., What Are the Hazards and Accident Experience From the Use of Pellet Powder?: Proc. Coal Min. Inst. America, 1938, pp. 49-52.

[52] Bureau of Mines Procedure for Applying for Tests Made on All Explosives and Blasting Devices by the Explosives Division of the Bureau of Mines; Prescribed Conditions and Requirements for Permissibility when Used in Coal Mines and Schedule of Fees for the Tests: Schedule 1C 1939, 13 pp. (Prepared by W. J. Huff and J. E. Tiffany.)

Float-and-sink data are best interpreted in terms of "washability curves."[53] These comprise four interrelated curves designated as "cumulative," "elementary," "specific-gravity," and "plus-and-minus 0.10 specific-gravity distribution" curves. All four curves have a common ordinate, so that values from one of the curves may be expressed in terms of any of the others. Assuming that the coal to which the curves apply has a size range suitable for wet table concentration, the curves can be used to predict the theoretical yield, average ash content, and highest ash content of particles in the washed coal. The efficiency of the separation is the ratio of the actual yield to the theoretical or float-and-sink yield and should be about 95 percent. It is not unusual for a table to operate at 97 to 98 percent efficiency.

A washability study of Woodstock bed coal[54] from the Cahaba field indicated that the "rash" problem is not serious for this coal. Proper operation of the washer, therefore, should facilitate removal of "rash" to an extent to minimize any adverse effects on the ash analysis. The study indicates that it should be possible to wash the minus 3-inch coal to approximately 4.4 percent ash with good efficiency, and that the coarser individual sizes of raw coal can be washed to a lower ash content than the fine sizes.

A washability study was made of Yolande No. 6 exploration slope, Warrior field, not yet positively identified as Black Creek bed coal, with particular reference to sulfur elimination.[55] The face sample showed higher sulfur content, somewhat higher heating value, and a lower ash-softening temperature than the usual Black Creek-bed coal. The sulfur percentage increased with increasing specific gravity of the float-and-sink portions; the ash-softening temperature showed no definite trend. If separate washing units are used for cleaning coarse and fine sizes, it should be possible to obtain washed coal of approximately 4.2 and 3.2 percent ash in the minus 3-inch plus 3/16-inch and the minus 3/16-inch sizes, respectively. The washed coal would contain about 1.6 percent total sulful - in 1:1 pyritic to organic form. This high ratio of pyritic to organic sulfur would appear to preclude the use of this coal for making metallurgical coke except when blended in small quantities with a low-sulfur coal. Flakiness data indicate that there is not enough flaky bone ("rash") in the coal to interfere seriously with reduction of ash in a properly designed washer.

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[53] Coe, G. D., An Explanation of Washability Curves for the Interpretation of Float-and-Sink Data on Coal: Bureau of Mines Inf. Circ. 7045, 1938, 10 pp.

[54] Gandrud, B. W., and Coe, G. D., A Washability Study of the Woodstock Coal Bed at Klondyke Mine, West Blocton, Ala.: Bureau of Mines Rept. of Investigations 3449, 1939, 12 pp.

[55] Gandrud, B. W., Coe, G. D., and Mead, J. C., A Washability Study of the Black Creek Coal Bed at Yolande No. 6, Rockcastle, Ala.: Bureau of Mines Rept. of Investigations 3450, 1939, 12 pp.

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Washability studies were made of the America and Pratt coal beds at Gorgas, Ala.[56] Face samples at three openings in the America and two in the Pratt bed were mixed for each bed in the proportions of the thickness of the bed at the individual points sampled. Washability curves indicated that the America bed and Pratt bed coals can be washed efficiently to approximately 9.1 percent and 7.6 percent ash, respectively. Proximate and ultimate analyses, heating values, forms of sulfur, and ash-softening temperatures were determined on combined standard seam samples from each bed.

Reduction of Washery Losses

The reduction of washery losses of marketable coal is a phase of coal preparation important to many Alabama coal producers. The practice at the Colta washery, the preparation of middlings for retabling, table tests on classifier overflow, and results of commercial plant operation and laboratory tests on Mary Lee-bed coal have been published.[57] The actual increase in yield of washed coal resulting from retreatment at the plant was 1.60 tons per hour, with a feed of 56 tons per hour to the primary tables. This is lower than the 2.18 tons per hour increase predicted from laboratory tests. The difference may be attributed both to the difference in character of the middlings re-treated in the plant and of those used in the laboratory tests, and to minor changes made in the operation of the primary tables so as to obtain a higher recovery of washed coal with a slightly lower ash content than that contemplated originally from the laboratory tests.

Washery-Slurry Study

The study of the washery-slurry-handling problem at coal preparation plants in Alabama has been continued. To avoid the many troubles arising from excess solids in the washery water it is necessary to withdraw some of the slurry in many of these plants from the washing system continuously, run it to waste, and replace it with clean fresh water. Data obtained in settling tests with and without flocculation with starch show that the capacity of settling cones could be increased enormously by the use of flocculation. This treatment solves the problem of water clarification but does not aid in thickening the settling sludge or dewatering the sludge after it settles out of the slurry. Froth-flotation tests have effected almost perfect separation of the coal from the ash-forming impurities. Recovery of the cleaned coal from froth flotation, however, involves the same settling and dewatering problems as recovery of solids from the original slurry. Preliminary tests indicate that a two-stage dewatering on screens, rather than the one-stage system now used at Alabama plants, should prove feasible. In the two-stage arrangement the underflow from the present dewatering screens would pass over fine-mesh, stainless-steel, vibrating screens, and the underflow from these would run back into the settling tank. This would give good dewatering of all coal coarser than the mesh of the vibrating screen and should result in less accumulation of solids in the washery water. The scheme has not been tried on a commercial scale.

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[56] Gandrud, B. W., and Coe, G. D., Washability Studies of the America and Pratt Coal Beds at Gorgas, Ala.: Bureau of Mines Rept. of Investigations 3458, 1939, 12 pp.

[57] Gandrud, B. W., Coe, G. D., and Hager, H. J., Classification and Tablings of Table Middlings at the Colta Coal Washery, Flat Creek, Ala.: Bureau of Mines Rept. of Investigations 3448, 1939, 13 pp.

Drainage Characteristics of Alabama Coals

A knowledge of the rate at which water drains from a given washed coal after it is loaded in railroad cars and during transit to the consumer is important for prediction of the moisture content of the coal following loading. The principal causes of the lack of uniformity in loss of water by drainage from different coals have been studied. The size distribution of the coal was found to be the most important factor affecting the rate of drainage.

A laboratory test has been developed that thus far appears to give drainage results comparable to those from railroad cars. The sample of coal is first soaked thoroughly for a definite length of time in a basket having a perforated-plate bottom. The basket is then weighed and allowed to drain for 24 hours while weighings are recorded at definite intervals. The percentages of moisture are plotted against time intervals on a logarithmic scale, and a straight line is drawn through the average of the co ordinate points. Results are reproducible within small limits of error. No readings are taken after 24 hours, but the straight-line graph is projected beyond the 24-hour point. Samples of coal taken from railroad car 3 to 9 days after loading had moisture contents that agreed within a few tenths of a percent with the corresponding values read from the projected graph. Predictions based upon the curve will be accurate under normal co ditions.

"Super Coal"

Float-and-sink separations were made on a seam sample of coal from Black Creek bed, Bradford mine, to determine whether any fraction could used as "super coal", that is, as a coal containing not more than 1 pe cent ash, suitable for use in internal-combustion motors. The coal was first stage-crushed through 28 mesh, and all minus 270-mesh material was rejected. The through 28-mesh and on 270-mesh material constituted 82.4 percent by weight of the coal and contained 3.6 percent ash. The float-1.30 specific gravity material was found to contain 1 percent ash and co prised 77.9 percent by weight of the material tested, or 64.2 percent of the original coal, thus showing that a high yield of "super coal" was po sible.

Drying and Storage Tests of Subbituminous Coals

Preliminary studies of the degradation of subbituminous coal during drying and storage for fixed periods and under known conditions of temperature and humidity have indicated the advantages of storage in sealed bins. Control tests included air-drying loss, proximate and ultimate analysis, heating value, screen analysis, and determination of friability.

Lignite Drying by Fleissner Process

The Fleissner process for drying lignite with high-pressure saturated steam has been studied further in a small pilot plant at the University of North Dakota. Approximately 50 tons of lignite from four large producing mines in North Dakota have been dried under various conditions, including changes in size of lignite, steam pressure, and cycles of heating. Normal tipple sizes of lignite have been studied to determine certain factors in the operation of the pilot plant, and the physical and chemical properties of the dried product have been determined as an aid in making an economic analysis of the process.

The amount of water removed during drying is a function of the steam pressure and is proportional to the increase in heating value. Some improvement in heating value also has resulted from removal of inert gases and from solution of part of the ash. Certain lignites respond differently from others to a given steam pressure, owing to differences in structure and composition. Those containing a relatively high proportion of woody material appear to dry in such a way as to give a better-appearing, even though more friable, product. The dried product has the peculiar property of repelling water; the surfaces do not remain wet after immersion, and water does not soak into the pieces.

UTILIZATION OF COAL

The efficiency of fuel utilization has increased strikingly compared with the disappointingly slow progress made in checking wastes during production.[58] In coal mining, destructive competition and the resulting unstable economic position of the industry tend to accentuate rather than minimize the waste of this resource. In the field of utilization, however, the same forces - competition and the necessity of profitable operation - act in a socially useful direction to promote economy in use.

A comprehensive analysis was made of the small consumer problem in selecting solid fuels.[59] Particular emphasis is placed on the burning characteristics of the fuel in the domestic appliances in which it is burned. The advice of reliable coal dealers and their combustion engineers on fuels whose properties have been well-established by properly conducted tests is of great service to small domestic consumers. New developments in the processing and carbonization of coal during 1938 were discussed in the annual review number of Mining and Metallurgy.[60]

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[58] Fieldner, A. C., Tryon, F. G., and Yancey, H. F., Mineral Fuels. Conservation in Use: House Document 160, 76th Congress, 1st session, Energy Resources and National Policy, part 3, sec. III, Conservation in Use, 1939, pp. 370-377. (Compiled by the National Resources Committee and other Federal Agencies.)

[59] Nicholls, P., Selection of Solid Fuels from the Viewpoint of the Small Consumer: Coal-Heat, vol. 35, February 1939, pp. 5-8; Heating, Piping, and Air Conditioning, vol. 11, February 1939, pp. 114-118.

Fuel-Economy Service

Upon request, this service advised Government establishments how to purchase fuel and equipment, conducts operating and acceptance tests on present and proposed heating and power-plant installations, and makes recommendations for their economical and efficient operation.

The Government was saved more than $82,000 as a result of careful analysis of requested appropriations for new equipment. Improvements in existing installations, comparisons of cost of operating with various fuels and introduction of improved methods of operating heating and power plants also have resulted in large savings to various Government agencies.

Such surveys were made for the Bureau of the Budget, Veterans' Administration, Department of Agriculture, Department of Justice, Office of Indian Affairs, United States Housing Authority, and the Procurement Division in 53 localities in the United States; for the District of Columbia and eight of its local power, heating, and sewage plants; and for the new Bureau of Mines station at Salt Lake City, Utah.

Boiler feedwater-conditioning service has been applied to 328 samples of water for local plants in Washington and 717 samples for eight Government Departments. Fourteen boiler compounds and 20 scales and sludges were analysed. An electric form of water treatment and a new type of compound for reducing corrosion have been tested for the War Department. Equipment was recommended and instructions were prepared for operating three water-analysis laboratories of the Government.

Smoke-Abatement, Fly-Ash, and Air-Pollution Control

Consulting service was given to various municipalities and Government Departments on smoke-abatement, fly-ash, and air-pollution problems.

The success of smoke abatement depends on human factors. Effective methods of attack depend on complete understanding by the public of all factors contributing to the nuisance. Operators must be trained in handling their particular fuel-burning equipment and filled with the idea of abating smoke, sulfurous gases, and fly ash. Devices for measuring and controlling these products are discussed in connection with effective city ordinances to give authority to this program.[61]

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[60] Fieldner, A. C., Processing and Carbonization of Coal: Min. and Met., vol. 20, January 1939, p. 50.

Restrictions on the emission of smoke, sulfur fumes, and fly ash have necessitated provisions for their control in coal-burning equipment.[62]

A Bureau of Mines exhibit showing improved methods of home heating and firing to prevent air pollution at the 33d annual (1939) convention of the Smoke Prevention Association at Milwaukee, Wis., attracted much interest and considerable favorable comment.

Current developments in methods of determining the source, composition, amount, and distribution of fly ash and in reducing or controlling its emission so as to eliminate or minimize the creation of a public nuisance have been published.[63] The papers contain about 30 illustrations of various methods of separating fly ash from the products of combustion.

A summary of published investigations, including a bibliography on the subject of sulfur compounds as damaging pollutants of air, reviews the type of sampling, analysis procedure, hour of day and time of year of sampling, number of locations, and number of determinations made in various cities of the United States and Europe.[64] Another summary of published investigations with a bibliography reviews the importance of sulfur compounds in the deterioration of materials as a basis for estimating the extent of property loss chargeable to such air pollution.[65] Materials included are cement, stone, metals, paint, leather, paper, and cloth. Both laboratory-type, in which the sulfur content and the humidity of the air are controlled, and field-type investigations, in which the exposure of materials to atmospheres are not under control, are described. A third summary of published data with bibliography reviews the distribution of sulfur from fuels when burned - that retained in the refuse, that lodging in the fuel-burning equipment, and that escaping into the atmosphere.[66] The effects of the composition of the ash-forming constituents in the fuel and of added chemicals in affecting the proportion retained in the refuse are summarized.

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[61] Barkley, J. F., Some Fundamentals of Smoke Abatement: Proc. 24th Fuel Eng. Conf., Appalachian Coals, Inc., Kalamazoo, Mich, Dec. 3, 1938, 5 pp.; discussion, 6 pp. (mimeographed); (more complete in) Manual of Ordinances and Requirements in the Interest of Air Pollution, Smoke Elimination, and Fuel Combustion, 1939, pp. 112-116; (and in) Bureau of Mines Inf. Circ. 7090, 1939, 59 pp.

[62] Barkley, J. F., Smoke and Fly Ash from Spreader Stokers: Combustion, vol. 9, No. 12, June 1938, pp. 37-38; Manual of Ordinances and Requirements of the Smoke Prevention Association in the Interest of Air Pollution, Smoke Elimination and Fuel Combustion: 1938, pp. 133-134.

[63] Barkley, J. F., Current Developments in Reducing Fly-Ash Emission from Chimneys: Proc. 33d Ann. Conv. Smoke Prevention Assoc., Milwaukee, Wis., June 13-16, 1939, pp. 161-173 (mimeographed), Manual of Ordinances and Requirements in the Interest of Air Pollution, Smoke Elimination, and Fuel Combustion, 1939, pp. 76-86, The Reduction of Fly Ash from Chimneys: Bureau of Mines Rept. of Investigations 3472, 1939, 10 pp.

[64] Burdick, L. R., and Barkley, J. F., Concentration of Sulfur Compounds in City Air: Bureau of Mines Inf. Circ. 7066, 1939, 14 pp.

[65] Burdick, L. R., and Barkley, J. F., Effect of Sulfur Compounds in the Air on Various Materials: Bureau of Mines Inf. Circ. 7064, 1939, 9 pp.

Viscosity of Coal-Ash Slags

The study of the viscosity of coal-ash slags was continued, using the oscillating bob viscometer and apparatus previously described.[67] The apparatus has proved to be satisfactory, and no major difficulties have been experienced in its operation. The preparation for and making of one test requires 2 to 3 days, but the tests made during the year were so distributed over the field of composition of coal ashes that some important general relationships could be established.[68]

Figure 8 is a plot of data of one typical slag. On the cooling cycle the solid phase started to form at the temperature of point A, and on the heating cycle the last crystals disappeared at point B; thus the liquidus temperature was about 2,280° F.

The major factors of composition to be considered in the study of coal-ash slags are five: Silica, alumina, lime-plus-magnesia, iron oxides, and the ferric percentage (ratio of ferric iron to total iron expressed as a percentage). The relations between these factors and the properties of the slags were found to be relatively simple.

1. For slags having an equivalent Fe_2O_3 content of 12 percent and lower there is little change in the liquidus temperature with a decrease in ferric percentage from its value in air to 10 percent; the viscosity may be increased up to 20 percent.

2. As the equivalent Fe_2O_3 increases above 12 percent, lowering the ferric percentage decreases the liquidus temperature until with 40 percent equivalent Fe_2O_3 the decrease is 400° F.; but again, there is little change in the viscosity of the liquid slag.

3. The viscosities of liquid slags decrease with increase in the percentage of lime-plus-iron oxide but are little affected by the ratio of lime to iron oxide.

4. Plots showed that as a first approximation the viscosity of the liquid slags is independent of the alumina content and, therefore, trilinear diagrams could be used.

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[66] Burdick, L. R., and Barkley, J. F., Sulfur in the Products of Combustion of Fuels: Bureau of Mines Inf. Circ. 7065, 1939, 23 pp.

[67] Fieldner, A. C., work cited in footnote 24, p. 15.

[68] Nicholls, P., and Reid, W. T., Viscosity of Coal-Ash Slags: Trans. Am. Soc. Mech. Eng., reprint, October 1939, 12 pp.

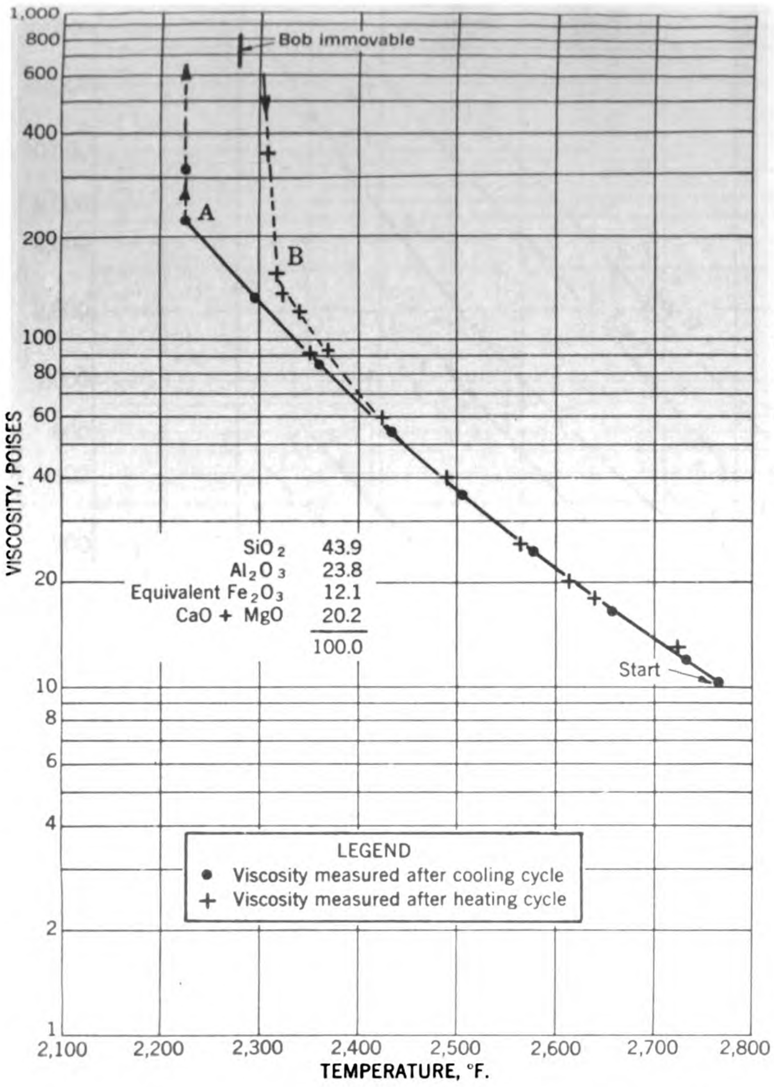

Figure 8.- Viscosity-temperature plot of a typical slag having an indefinite liquidus temperature.

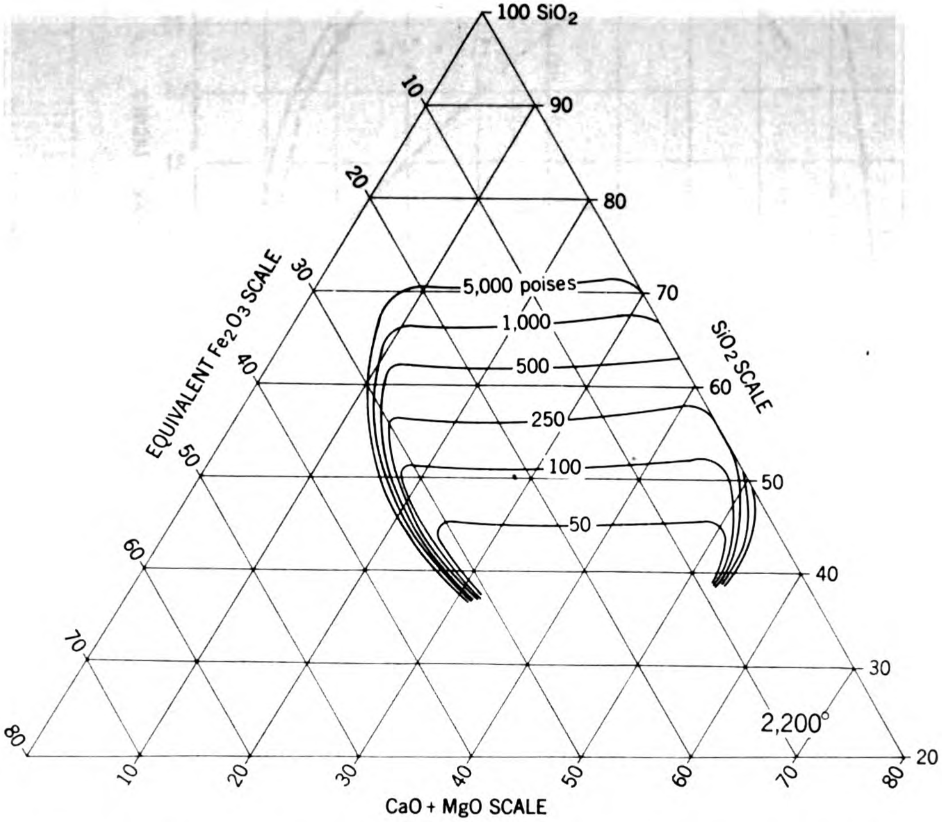

Figure 9.- Suggested form of complete plot of viscosity for a fixed silica-alumina ratio and a fixed ferric percentage.

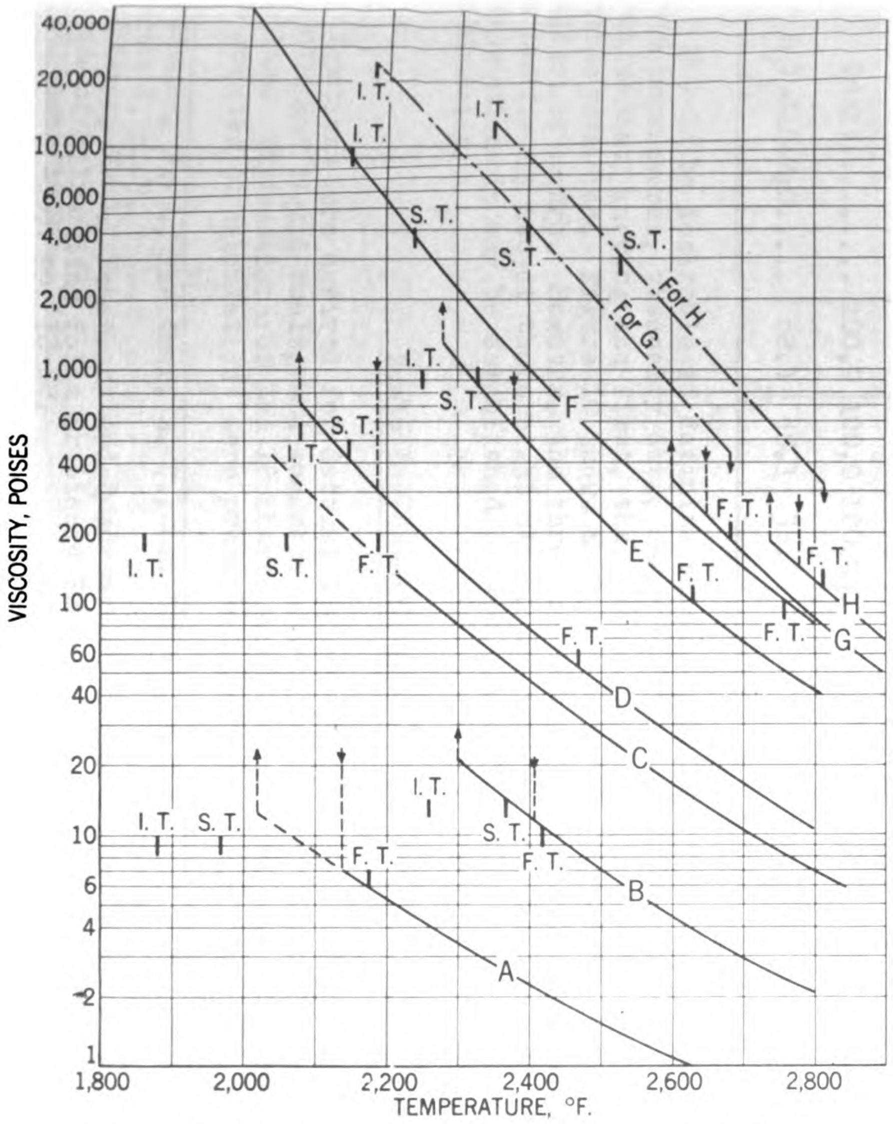

Figure 10.- Cone-fusion temperatures and viscosity curves for eight coal-ash slags.

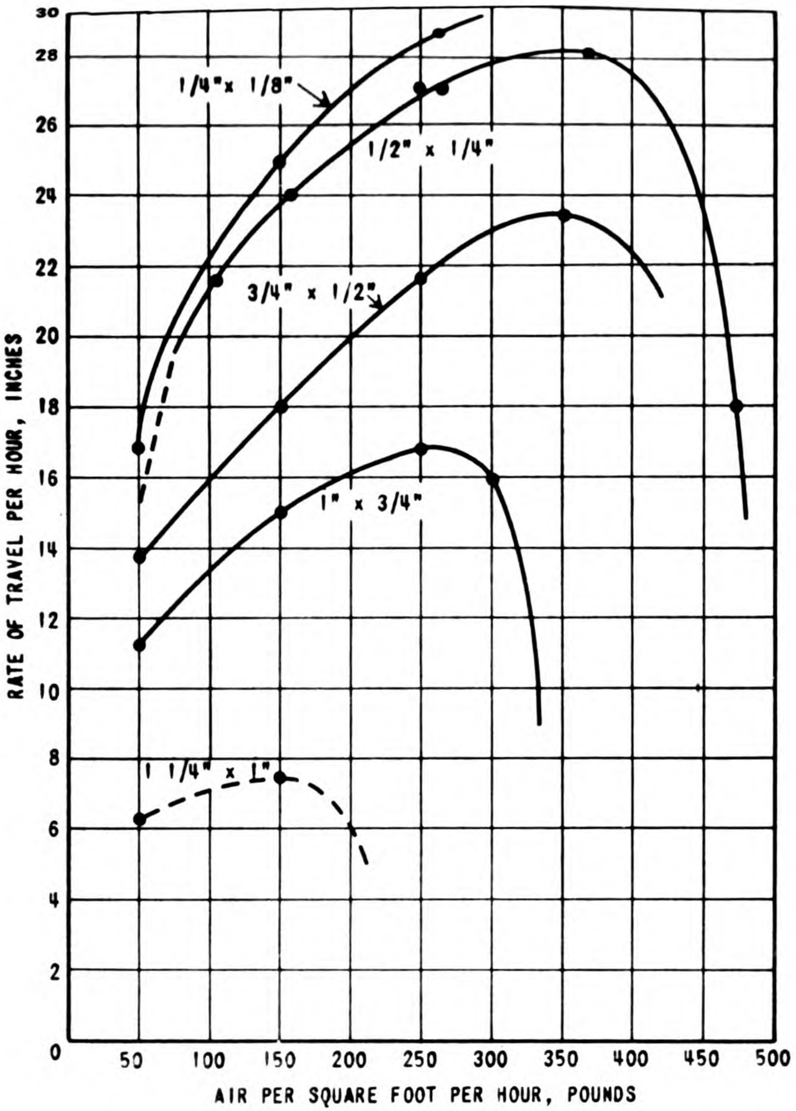

Figure 11.- Rate of travel of ignition of high-temperature coke.

5. Figure 9 suggests the type of plot that would give complete data on the fusibility of coal ash; one such plot would be required for each temperature, a fixed silica-alumina ratio, and a fixed ferric percentage.

Figure 10 shows the cone-fusion temperatures and viscosity data for eight coal-ash slags having the compositions given in table 1. The last line of the table gives the total flux. The curves of figure 10 lie in higher ranges of viscosity as the total flux decreases; the abbreviations I.T., S.T., and F.T., shown for each slag (not the original coal ash), are, respectively, initial deformation temperature, softening temperature, and fluid temperature observed in the cone-fusion determination.[69]

TABLE 1. - Composition of coal-ash slags, weight percent

Constituents	Coal-ash slags							
	A	B	C	D	E	F	G	H
SiO_2..............	26.3	35.3	41.4	48.4	50.0	57.5	53.3	55.3
Al_2O_3..............	11.6	16.6	22.3	19.2	26.7	19.1	28.3	31.1
Equivalent Fe_2O_3..	46.1	9.9	31.4	12.1	13.1	13.2	13.7	9.5
CaO+MgO...........	16.0	38.2	4.9	20.3	10.2	10.2	4.7	4.1
Totals.........	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Fe_2O_3+CaO+MgO.....	62.1	48.1	36.3	32.4	23.3	23.4	18.4	13.6

It would be desirable if definite relations between the three cone-fusion temperatures, viscosity data, and liquidus temperature could be found, so that, knowing the first, the viscosity and liquidus temperature could be predicted. Figure 10 shows that the softening temperature alone would not be enough. However, the three cone-fusion temperatures are not the same for any two of these eight slags, and thus it is possible that some relationship may be established between the cone-fusion temperatures and viscosity.

Fuels for Ceramic Kilns

Equations were derived for determining heat balances of kilns utilizing solid and liquid fuels, and quadrant charts were constructed for graphic solutions. Explanations of the 10 charts and 21 equations with illustrative examples of calculations were presented.[70]

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[69] Stanton, F. M., Fieldner, A. C., and Selvig, W. A., work cited in footnote 8, p. 7.

[70] Skinner, K. G., Equations and Quadrant Charts for Determining Heat Balance of Kilns Using Solid or Liquid Fuels: Jour. Am. Ceram. Soc., vol. 22, March 1939, pp. 72-82.

The selection of suitable coal for ceramic kilns and the process used or, conversely, the adaptation of the design of furnaces and the method of burning coal in them to suit the coals that are available have been studied.[71] Equipment and procedure of tests, coals used, and design of furnaces are described in relation to test data obtained, and general conclusions are based on these data.

Burning Characteristic of Anthracites

The burning characteristics of six anthracites covering a wide range of volatile matter from different districts in two of the fields of Pennsylvania were determined for carefully rescreened commercial sizes in a special hand-fired (overfeed) furnace at domestic rates under imposed controlled conditions.[72] Brief conclusions show that (1) if the physical characteristics of the anthracites and the fusion temperatures of the ash are approximately the same, the ease of pick-up increases with increase of volatile matter and the average rate of burning is affected little by volatile content, except that differences in pick-up lengthen or shorten the time required to reach a steady state of burning; (2) large differences in quantity of ash or its fusion temperature affect the burning characteristics; (3) the physical characteristic of anthracites that most influences the burning characteristics is whether the piec when heated retain their size or split or break into smaller pieces; and (4) if the pieces break up in the fuel bed the burning characteristics correspond to those of a fuel of a smaller size than that fired.

Combustion on Overfeed and Underfeed Domestic Stokers

The overfeed stoker tests described in the report of last year[73] have been continued. Results of tests on coals from the Southport and Alpine mines of the Coos Bay coal field have been published.[74] The equipment used was the same as that referred to last year. Two tests were made on each coal - one with the stoker feed-gate set for intermediate rate and the other at maximum rate. Owing to the coarse size of the two coals, however, the intermediate-rate test on each coal corresponded to the minimum-feed rate with other coals previously tested in smaller sizes. Over-all efficiencies of 78.2 and 69.5 percent were obtained on the overfeed stoker-boiler combination with the Southport coal when 12.1 and 21.5 pounds of coal were burned per hour, respectively. Alpine coal had an over-all efficiency of 74.9 and 71.3 percent at burning rates of 12.6 and 21.7 pounds of coal per hour, respectively. The lower efficiency of 69.5 percent for the Southport coal at its maximum rate of burning, 21.5 pounds of coal per hour, was due to too small excess of air supply. The chief difference in behavior of the two coals was in the character of the ashes produced. Southport coal had an ash-softening temperature of 2,170° F. and formed larger, more completely fused glassy clinkers than the Alpine coal, which had an ash-softening temperature of 2,700° F. and formed smaller friable clinkers with much porous ash. The results showed that both coals could be used efficiently for house-heating purposes with equipment like that used in the tests.

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[71] Rice, W. E., and Austin, C. R., Burning of Coal in Down-Draft Ceramic Kilns and Burning Characteristics of Some Ohio Coals: Bureau of Mines Tech. Paper 598, 1939, 34 pp.

[72] Nicholls, P., and Landry, B. A., Burning Characteristics of Anthracites: Trans. 2d Ann. Anthracite Conf., Lehigh University, Apr. 28-
1939, pp. 11-26.

[73] Fieldner, A. C., work cited in footnote 24, p.15.

[74] Yancey, H. F., Stoker Tests on Coos Bay Coal by Northwest Experiment Station, Federal Bureau of Mines, Seattle, Wash.: "Progress Report on Coos Bay Coal Field," Oregon State Dept. Geol. and Mineral Industries, Bull. 2, 2d ed., May 1939, appendix, pp. 14-15.

During the fiscal year 14 burning trials were made on the overfeed and 20 on the underfeed stoker-boiler combination with coals ranging in rank from medium-volatile bituminous to lignite. Because ashes may be removed from the ashpit in the usual way, the overfeed stoker is suitable for burning low-rank, noncaking coals of low-, medium-, and especially high-ash contents. The underfeed stoker is desirable for both caking and noncaking coals, but because clinkers must not be allowed to build up too much in the fuel bed, coals of low ash content are preferable. High over-all efficiencies have been obtained with both types of stokers at burning rates up to 20 pounds of coal per hour; 75 percent efficiency is common. Ordinarily, low-rank subbituminous coals and even lignites may be burned nearly as efficiently as the higher-rank coals, but the heat output is less in proportion to the reduction in heating value.

The possibilities of both static and dynamic pressures must be considered in the interpreting pressure data obtained in tests of underfeed stokers in power plants. Similarly, the meaning of gas-analysis data is definite only if the air rate is constant and the contour of air flow in relation to that of the fuel bed from front to back is known. Consideration must be given to feed conditions and distribution of the coal in the fuel bed.[75]

Combustion on Traveling Grates

The burning of solid fuels under conditions closely simulating those prevailing in fuel beds on large traveling grates is being investigated.

The furnace consists of a firepot lined with a 5-inch thickness of light-weight refractory and provided with a 20-inch square grate with a possible maximum fuel-bed thickness of 8 inches. The firepot is of welded construction with two flanged openings to the ashpit, one for the door and one for connection to the supply of air. A flue on one side leads to a short water-cooled stack, from which gas samples are drawn continuously during a test. Two interchangeable tops are hung from corner posts in such a manner that they may be quickly swung over the furnace and lowered into a mercury seal.

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[75] Nicholls, P., Discussion of "The Fuel-Bed Tests at Hell Gate Generating Station" by M. A. Mayers: Proc. 23d Fuel Eng. Conf., Appalachian Coals, Inc., Cincinnati, Ohio, May 5, 1938, 4 pp. (mimeographed).

The "ignition" top contains six globars set in a refractory. The whole cover is gas-tight when it is in place over the furnace. When the "ignition" top is in its off position it faces a water-cooled plate, which absorbs some of the radiated heat while the top is being heated. Power is supplied to the ignition top so as to give a radiating temperature of 2,400° F. under the conditions of the test. For high-temperature coke at high air-supply rates, 2,300° F. is a safer rating. Two porcelain-protected rare-metal thermocouples are so placed in the ignition top as to give an average of the radiating temperature of the globars and refractory surfaces. The other cover is refractory-lined, with a water-cooled chimne in its center. When it is in place over the furnace the side stack of the furnace is closed. When this "refractory" top is in its off position it forms the cover of a gas-fired furnace, which can heat it to any desired temperature before it is brought over the firepot.

The primary air is supplied by a motor-driven fan with an available pressure head of 12 inches. The air passes through a special (central-opening, regulating) damper, an orifice, a preheater, and then to the furnace. All joints are air-tight, and close regulation of the air is possib

Temperatures in the fuel bed are measured by thermocouples placed at known heights - one on the top, one on the grate, and others at intermedia heights.

To carry out a test, the ignition top is raised to temperature when in its off position, and the time at which it is quickly transferred to the charged furnace marks the beginning of the test. The fan is started and the air-supply rate broughtto that selected for the test. The fuel bed is ignited by radiation from the ignition top. As the temperature of the bed rises during down-burning of the fuel, the power needed by the "ignition" top to maintain its temperature decreases. With some fuels and air rates the heat radiated by the bed becomes greater than that of th top, and power required to maintain its temperature become zero. This fixes the time when the ignition top is replaced by the preheated refractory top. When the heat radiated by the bed is not enough to reduce the power requirements of the ignition top to zero, the time of changing the tops is fixed at some other condition, such as the plane of ignition having reached a certain depth downward from the top of the fuel bed. Other criteria for fixing this time are when the carbon dioxide indicator shows the maximum content of this gas in the stack and visual observation of the fuel bed through a peephole in the furnace. Flue gas is drawn fro the stack continuously, sampled, and analyzed.

The tests to date have been with closely sized coke. Figure 11 is a plot of the rate of travel of the plane of ignition downward through the fuel bed against the rate of air flow. The rate of travel of ignition fo each size increases with air-flow rate to a certain maximum and then falls rapidly until combustion cannot be maintained. The limiting rate of flow of air increases with decrease in the mean size of the coke. However, as the size of the coke decreases, the air rate at which the fuel bed begins to "boil" decreases, namely, when excessive fuel is lifted from the bed. This action destroys the uniformity of the bed, and results are less reliable. Thus, the tests with the 1/4-inch to 1/8-inch size could not be carried beyond an air rate of 250 pounds per square foot of grate area per hour.

Coke as a Domestic Fuel

The good features of coke as a domestic fuel and recommendations for most efficient and economical firing practices were discussed.[16]

Properties of Western Cokes

A comparative investigation of the physical and chemical properties of commercial cokes produced or used in Washington has been made by the Northwest (Seattle, Wash.) Experiment Station, in cooperation with the College of Mines, University of Washington.[17]

The physical and chemical properties of 14 cokes from Washington, 3 from British Columbia, and 1 each from Utah, Pennsylvania, England, and Germany were determined by standard methods of test. All cokes were produced on an industrial scale by commercial processes. Eleven cokes were beehive, 5 byproduct, 3 retort, and 2 low-temperature. The foreign cokes were included for comparative purposes because small amounts reach the ports of Washington. The sample from Pennsylvania, a low-temperature coke, was used for reactivity tests only.

Tests on the 20 other cokes included proximate analysis, heating value, sulfur, phosphorus, softening temperature of the ash, apparent and true specific gravity with calculated cell space, shatter and tumbler with calculated friability factors, reactivity, and ignition-temperature determinations. The first four determinations listed and the agglutinating-value test were made on 17 of the corresponding coals used to produce these cokes. Samples of the coals from England and Germany and of one coal from Washington were not obtainable. The same four determinations, in part, were carried out on the Pennsylvania low-temperature coke, a willow charcoal, a Washington weathered subbituminous coal, a Washington bituminous coking coal, a Pennsylvania anthracite, and an electrode carbon. These latter samples were used for reactivity tests only for comparison with reactivity results obtained on the 20 commercial cokes.

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[16] Nicholls, P., Coke as a Domestic Fuel: Coal-Heat, vol. 34, December 1938, pp. 32-33, 44.

[17] Yancey, H. F., Zane, R. E., Fatzinger, R. W., and Key, J. A., Physical and Chemical Properties of Cokes Made or Used in Washington: Bureau of Mines Tech. Paper 597, 1939, 44 pp.

Reactivity in air was measured by two methods. One was a modification of the method used by Kreulen, and the other was a new method develope for the work. The modified Kreulen method has several disadvantages that prevent satisfactory comparison of results on samples covering a wide range of reactivity. The method makes use of too small a sample to complete the test with materials of high reactivity, such as charcoal. Moreover, the samples are of such fine size (100- to 200-mesh) that surface and porosity characteristics are destroyed to a considerable extent, thus affecting seriously the nature and extent of combustibility. Furthermore, the surface of the coke sample presented to the action of air during the test has about three times the area of the top dimensions of the porcelain boat used to hold the sample. Since this latter area is employed in the method of calculating the "reactivity index," that is, the number of milligrams of carbon dioxide formed in 5 minutes per square centimeter of area at 600° C. the "reactivity-index" value appears much larger than it really should be. A similar argument applies to "ignition temperature," defined arbitrarily as that at which 10 milligrams of carbon dioxide are formed in a 5-minute period per square centimeter of area of coke exposed. The reactivity indexes obtained by the modified Kreulen (oxidation with air) method show only rough qualitative agreement with those given by the Bureau of Mines (reduction of carbon dioxide) method.

To overcome the disadvantages cited, a new combustion-rate method was developed. This method consisted in measuring simultaneously the weight-loss in percentage and time in minutes of a 1-gram sample of sized coke or coal (through No. 8 and on No. 12 of the U. S. sieve series) during its burning in air (1.75 liters per minute) in a 40-mesh, nichrome-wire basket, which is suspended inside an electrically heated, standard Fieldner volatil matter furnace from a specially constructed balance. The air stream was broken up and distributed before it reached the basket by passage through a nichrome-wire screen. A thermocouple whose hot junction was at a point within half an inch of the loaded basket measured the reaction temperature.

Combustibility results obtained by this new method were expressed in cumulative weight-loss percentages, at 5-minute intervals, at 500°, 600°, 700°, 800°, and 900° C. reaction temperature. Plots of the cumulative weight-loss versus time for the various samples at each temperature gave curves whose slopes were measures of the relative rates of burning of the various samples for the given reaction temperature. With increasing reaction temperatures all samples showed greater combustion rates, although the magnitude of the increased rate was not the same for each sample. An improved method of expressing the results of combustion tests employs the so-called "50-percent index," namely, the time, in minutes, required to burn the sample to half its original weight. During this period, effects such as differences in the ash content of individual samples have time to modify the results. From the 50-percent indexes the corresponding combustion rates of the cokes may be calculated directly. These rates fall in an order the reverse of that of their respective 50-percent indexes, but are more directly applicable for comparing the relative combustibility of various cokes.

Although the modified Kreulen method and the new combustion-rate method place the cokes in essentially the same order, the latter is superior in that the test samples are more representative of the coke investigated, the results obtained are more concrete, and the test simulates the actual use of the coke.

Washington cokes, especially the byproduct and retort cokes, were found to be fairly reactive. The two low-temperature cokes (one from Washington and one from Pennsylvania) were more reactive, approaching the coals from which they were made. At high temperatures, all cokes were equally active and all seemed to burn at the same rate. Results obtained by the combustion-rate method on two series of cokes produced at 500°, 600°, 700°, 800°, 900°, and 1000° C. in the BM-AGA retort from Lower Sunnyside bed (Utah) high-volatile B bituminous coal and from Beckley-bed (W. Va.) low-volatile bituminous coal showed that average combustion rates for the cokes made from the lower-rank (Utah) coal were generally higher than for the cokes made from the higher-rank (W. Va.) coal. However, higher combustion rates, especially at 600° and 700° C. reaction temperatures, are given by cokes from the West Virginia coal when carbonized at 900° and 1,000° C. Since low- and medium-temperature cokes made from high-volatile or low-rank coals contain relatively more volatile matter than similar cokes produced from low-volatile or high-rank coals the combustion rates of the former are higher. At high carbonization temperatures the volatile-matter content of various cokes does not vary appreciably, and their rates of combustion are virtually alike.

The ignition temperature of Washington cokes, obtained by the modified Kreulen method, ranged from 460° to 570° C., the higher temperatures being for the Wilkeson cokes. The low-temperature cokes and willow charcoal have lower ignition temperatures. Ignition temperatures decreased with increasing volatile-matter content.

Because of the wide differences not only in the rank of the coals made into coke but also because the carbonization processes were different, no attempt was made to correlate the reactivity and ignition characteristics with the specific chemical and physical properties of the cokes.

All Washington cokes have a rather high ash content. Adoption of improved methods of coal preparation probably will permit the production of coke of lower ash content.

Results of physical tests on 20 commercial cokes produced in Washington and elsewhere, when compared on the same basis by the standard testing methods for evaluating the various properties of coke, show that a number of the Washington cokes compare favorably with those made elsewhere.

Standard shatter tests on the coke samples demonstrate that strong cokes, resistant to shatter degradation, are made from Washington coals. Wilkeson cokes from Pierce County coal have an average 2-inch shatter index of 82, which is higher than that of many eastern, southern, and midwestern cokes.

The beehive cokes from the Wilkeson area have high stability factors (cumulative percentages remaining on 1.05-inch screens); that is, they are resistant to the breaking effect of the combined attrition and impact in the tumbler test. Lower stability is shown by the byproduct cokes from Washington and British Columbia. Utah coke had an extremely low stability index, even lower than that of the low-temperature coke from Washington. Smaller differences are shown by the hardness factors (cumulative percentages retained on a 0.263-inch sieve). Utah coke had the highest hardness factor, 68.2; evidently, it is resistant to abrasion, though not to impact. Stability and hardness factors of the Wilkeson beehive coke increased with increase in carbonization time.

The "percentage friability," calculated from all sizes in the whole screen analysis of either the shatter or tumbler tests, shows average percentage degradation in size instead of the percentage remaining on individual screens. On the basis of friability, calculated from the tumbler-test results, the cokes are grouped in the following order: Wilkeson beehive, 58.9 to 68.5; Washington byproduct and retort, 73.8 to 77.0; Utah byproduct, 79.9; Washington low-temperature Coalene, 86.1; British Columbia byproduct, 55.8; British gas-chamber, 76.9; English byproduct, 42.6; and German byproduct, 39.3 percent.

Tumbler tests for a number of eastern and southern cokes were compared with those reported in the literature. Washington cokes are more resistant to breaking into small pieces but less resistant to abrasion.

Utilization of Coal-Mine Refuse

Coal-mine refuse from Mary Lee and Corona coal beds in Alabama have been investigated as to their suitability for industrial materials. The proposed uses thus far studied are (1) as an activated char for water-purification purposes; (2) a decolorizing agent for mineral oils; (3) a base exchanger for water softening; and (4) a soil conditioner for agriculture. Instead of using the coal refuse as a whole, it was found more practicable to fractionate, by tabling, into a number of specific-gravity fractions.

In preparing an activated char, the refuse was first charred at 500° C. in a closed retort, then ground to a fine powder, and finally acti vated with steam at 750° to 950° C. in a large quartz tube. The optimum conditions for carrying out these operations varied with the quality of the original refuse. The degree of activation of each product was measure by the phenol absorption test. The results obtained indicated that the lower the ash of the refuse, the more active the char as measured by the phenol number. The mineral matter in the refuse acted as a diluent and no test made thus far has shown any evidence of its activation. Activati with zinc chloride instead of steam (the excess being washed out by means of strong hydrochloric acid followed by water) also indicated promising results.

Contact clays activated by means of chemicals are one type used for decolorizing mineral oils. A study of the composition, together with the effect of various heat treatments, of different specific-gravity portions of coal refuse showed that the high-ash fraction responded most favorably to chemical activation. The best result was obtained by treating the high-ash portion of Mary Lee-coal refuse with a dilute solution of ammonium hydroxide, followed by a dilute wash with phosphoric acid in the presence of a salt, such as aluminum phosphate. This treatment gave a product that decolorized oil about 75 percent as well, at room temperatures, as a good commercial activated clay. At higher temperatures, however, the activated coal refuse lost most of its effectiveness, whereas the effectiveness of commercial clay increased. All attempts thus far to improve the activity of the coal refuse above room temperature have been unsuccessful.

The experimental work on coal-mine refuse as a base exchanger for water softening has clearly indicated that both the clay and the coal in the refuse have base-exchanging properties, but that the coal was the better of the two. High-coal fractions of the refuse were digested with sulfuric acid at 100° C. The acid-treated product (hydrogen base exchanger) was then converted with dilute alkali under a steam pressure of 11 atmospheres into a sodium base exchanger. Corona-coal refuse, containing 11 percent ash and 89 percent fixed carbon and volatile matter, gave a sodium base-exchange capacity of 5,500 grains per cubic foot compared with 3,000 grains per cubic foot for Mary Lee coal refuse containing 19.5 percent ash and 79.5 percent fixed carbon plus volatile. These capacities are for zero hardness water. Part of the difference in capacity may be attributed to the variation in the coal content, and part, perhaps, to the nature of the coal itself. The hardness of the raw or test water used was 30.5 grains per gallon.

Artificial darkening of a light-colored soil by addition of black-coal refuse (1) to increase absorption of the sun's rays and consequently maintain a higher temperature to promote growth of crops in early spring and (2) to decrease reflection of the sun's rays by the soil to eliminate tip burning of certain plants are interesting agricultural possibilities. Coal refuse is being investigated as a potential source of available humic acid to serve as a fertilizer for soils.

Survey of Carbonizing Properties of American Coals

Results of the investigations of the BM-AGA survey of the carbonizing properties and petrographic composition of 8 coals (numbered 47 to 54, inclusive, in the survey), 14 blends prepared from these and two blending coals, and 6 oxidized coals were described briefly last year.[78] Publications covering the details of these investigations have been issued.[79]

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[78] Fieldner, A. C., Work cited in footnote 24, p. 15.

The survey was continued during the present fiscal year and has included work on 6 coals (designated 55 to 60, inclusive), 8 blends, and 2 blending coals.

Laboratory low- and high-temperature carbonization assays were made on coals 55 to 59, and agglutinating values were obtained for the same coals and their 10 blends.

Table 2 gives the sources of the six coals and of the two coals used with them in blend tests, together with the ranks, analyses, and heating values of these 10 coals and of the 8 blends tested. Coals 55, 58, and 60 are medium-volatile and coals 56 and 57 low-volatile bituminous coals. Coals 28 and a36, used in preparing the blends, are high-volatile A coals. Coal 59 is high-volatile A, but high in that range of rank. All these coals are classed as bright; however, the Alma (a36) coal contains 25 percent splint and 14 percent semisplint coal. All contain less than 9 percent ash (dry basis), and their sulfur content is such that they produce coke containing 1.3 percent or less sulfur; they are of suitable grade therefore, for manufacture of blast-furnace coke. Consistent with their rank, the medium- and low-volatile coals give high yields of blocky coke and moderate yields of byproducts. Coals 55, 56, 57 and 60 are strongly expanding. Although coals 58 and 59 showed contraction in the expansion test ovens (see table 8) they deformed the BM-AGA retorts, and caution probably should be used in charging them alone in a coke oven.

Table 3 gives the yields of carbonization products obtained for coals 55 to 58 inclusive, for blending coals a36 and 28, and also for the blends. The table shows that coals 55, 56, 57, and 58 give low yields of tar and light oil and high yields of coke compared with high-volatile blending coals 28 and a36 and the blends in which 70 and 80 percent of coals a36 and 28 were used. The differences in yields of gas on the volume basis ar not large, but on the basis of heating value of gas per pound of coal carbonized it is clear that the high-volatile coals and blends have a decided advantage. These results are consistent with differences in rank of the coals as measured by the proximate analysis. The more volatile matter liberated during the laboratory analysis, the more volatile carbonization products are to be expected when allowance is made for differences in

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[79] Fieldner, A. C., Davis, J. D., and others, Carbonizing Properties and Petrographic Composition of Washed and Unwashed Lower and Upper Kittanning-Bed Coals from Mines 72 and 73, Johnstown, Cambria County, Pa. Bureau of Mines Tech. Paper 595, 1939, 83 pp.
Davis, J. D., and Parry, V. F., Carbonizing Properties of a Subbitumi
Coal from Puritan Mine, Dacono, Weld County, Colo.: Bureau of Mines Rept. of Investigations 3428, 1939, 32 pp.
Fieldner, A. C., Davis, J. D., and others, work cited in footnote 18,
Fieldner, A. C., Davis, J. D., and others, Carbonizing Properties and Petrographic Composition of Pond Creek Bed Coal from Majestic Mine, Majestic, Pike County, Ky.: Bureau of Mines Tech. Paper 596, 1939, 4
Fieldner, A. C., Davis, J. D., and others, work cited in footnote 19,

TABLE 2. - Analyses of coals, as carbonized[1]

Coal 55 - Sewell bed, Wyoming mine, Wyoming County, W. Va.
Coal 56 - Pocahontas No. 3 bed, Buckeye No. 3 mine, Wyoming County, W. Va.
Coal 57 - Pocahontas No. 4 bed, No. 4 mine, Raleigh County, W. Va.
Coal 58 - Lower Banner bed, Keen Mountain mine, Buchanan County, Va.
Coal 59 - Upper Freeport bed, No. 21 mine, Monongalia County, W. Va.
Coal 60 - Lower Freeport bed, No. 9 mine, Indiana County, Pa.
Coal a36 - Alma bed, Red Jacket mine, Mingo County, W. Va.
Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.

Coal No.[2]	Dry, mineral-matter-free, fixed carbon	Moist, mineral-matter-free B.t.u.	Proximate, percent				Ultimate, percent					Heating value, B.t.u. per lb.
			Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur	
55	77.5	15,450	1.6	21.8	73.7	2.9	4.9	85.8	1.4	4.3	0.7	14,950
55A	63.5	15,230	1.9	34.6	59.5	4.0	5.4	81.8	1.4	6.9	.5	14,550
55B	65.4	15,220	1.9	32.9	61.4	3.8	5.4	82.3	1.5	6.4	.6	14,570
56	81.7	15,520	1.6	17.4	74.5	6.5	4.4	83.7	1.2	3.6	.6	14,400
56A	66.0	15,150	1.6	32.0	60.8	5.6	5.2	79.8	1.5	7.0	.9	14,200
56B	67.8	15,140	1.6	30.3	62.3	5.8	5.1	80.2	1.4	6.6	.9	14,160
57	83.3	15,510	1.4	16.0	75.6	7.0	4.3	82.6	1.3	3.7	1.1	14,300
57A	66.4	15,130	1.5	31.6	61.2	5.7	5.2	80.3	1.5	6.3	1.0	14,170
57B	68.2	15,200	1.6	29.9	62.7	5.8	5.1	80.7	1.5	5.9	1.0	14,210
58	76.9	15,390	2.5	21.4	67.8	8.3	4.7	79.5	1.2	4.8	1.5	13,960
58A	64.9	15,180	1.7	32.9	59.4	6.0	5.3	79.5	1.5	6.7	1.0	14,160
58B	66.0	15,140	1.8	31.8	60.3	6.1	5.2	79.1	1.4	7.0	1.2	14,110
59	67.7	15,080	2.8	29.3	59.6	8.3	5.2	77.0	1.4	7.4	.7	13,690
60	73.5	15,400	2.0	24.9	66.7	6.4	5.1	81.2	1.3	5.3	.7	14,310
a36[3]	60.4	15,190	1.7	37.5	56.5	4.3	5.5	80.8	1.4	7.5	.5	14,460
28[4]	61.8	15,080	1.5	36.0	57.3	5.2	5.4	79.6	1.6	7.2	1.0	14,200

[1] Analyses by H. M. Cooper, chemist, Bureau of Mines.

[2] Coal 55A contains 80 percent and coal 55B contains 70 percent Alma bed coal (No. a36); coals 56A, 57A, and 58A contain 80 percent and 56B, 57B, and 58B contain 70 percent Pittsburgh bed (Warden mine) coal (No. 28).

[3] Resampled July 16, 1938, and used in blending with coal 55.

[4] Used in blending with coals 56, 57, and 58.

TABLE 3. - Yields of carbonization products obtained at 900° C. in the 18-inch retort

Coal 55 - Sewell bed, Wyoming mine, Wyoming County, W. Va.
Coal 56 - Pocahontas No. 3 bed, Buckeye No. 3 mine, Wyoming County, W. Va.
Coal 57 - Pocahontas No. 4 bed, No. 4 mine, Raleigh County, W. Va.
Coal 58 - Lower Banner bed, Keen Mountain mine, Buchanan County, Va.
Coal a36 - Alma bed, Red Jacket mine, Mingo County, W. Va.
Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.

		Yields per ton of coal					
Coal No.[1]	Coke, percent	Gas, cubic feet	Tar, gallons	Light oil in gas, gallons	$(NH_4)_2SO_4$, pounds	Liquor, percent	B.t.u. per pound of coal
55	80.0	10,650	6.9	1.77	16.1	3.4	2,930
55A	71.3	10,850	12.8	3.15	17.5	4.0	3,310
55B	72.1	10,650	12.9	3.25	18.2	4.2	3,300
56	84.0	10,450	4.1	1.36	13.4	2.7	2,610
56A	72.1	10,550	12.8	2.57	21.8	5.5	3,120
56B	73.9	10,450	11.9	2.69	19.2	5.2	3,030
57	84.3	10,250	3.2	1.29	14.5	2.4	2,590
57A	72.4	10,650	12.5	2.85	21.0	5.6	2,970
57B	74.0	10,500	11.1	2.64	20.1	5.3	3,060
58	79.2	10,150	6.4	1.92	15.2	4.9	2,870
58A	71.2	10,450	13.3	3.18	22.4	5.7	3,140
58B	72.3	10,450	12.2	2.95	22.1	5.4	3,150
a36[2]	68.5	11,050	14.2	3.04	17.4	5.9	3,450
28[3]	68.8	10,650	14.9	2.79	19.0	6.4	3,230

[1] Coal 55A contains 80 percent and coal 55B contains 70 percent Alma bed coal (no. a36); coals 56A, 57A, and 58A contain 80 percent and 56B, 57 and 58B contain 70 percent Pittsburgh bed (Warden mine) coal (no. 28).

[2] Resampled July 16, 1938, and used in blending with coal 55.

[3] Used in blending with coals 56, 57, and 58.

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carbonizing conditions between the volatile matter and carbonization tests. These medium- and low-volatile coals yield little ammonium sulfate. The reason for this is not that they contain less nitrogen than do the high-volatile coals and blends (see table 2), but that the two latter give comparatively high yields of liquor, and it is known that yields of ammonia are improved by carbonization in the presence of water vapor.

Table 4 gives the shatter and tumbler indexes, which indicate that the cokes from these medium-volatile and low-volatile coals are among the strongest tested in the BM-AGA survey. The blend cokes from charges containing 80 percent high-volatile coal invariably are weaker than those from the unblended charges, but when the proportion of high-volatile coal is reduced to 70 percent in the blends, the strength of the coke approaches closely that of coke from 100-percent low- or medium-volatile coal. The 100-percent charges deformed the BM-AGA retorts more or less, and therefore coking must have taken place under pressure for at least a part of the coking period. One might expect the coke to be compacted somewhat by this pressure. As a matter of fact, the apparent specific gravity of these cokes is higher than that of the blends, with which deformation of the retorts did not occur.

The four completed investigations on the coals and blends tested in the BM-AGA survey during the fiscal year have been published,[80] are in press[81] or in manuscript form.[82]

Composition of Coal Tar and Light Oil

Bulletin 412, Composition of Coal Tar and Light Oil, reviewing the published information and including a selected bibliography on the chemical and physical properties of light oil and coal tar produced under both low- and high-temperature conditions, has been reprinted.[83]

Effect of Conditioning in Coking Properties of Coal

Representative samples from seven coal beds were subjected to accelerated weathering tests in air at 99.3° C. in the apparatus described

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[80] Fieldner, A. C., Davis, J. D., Selvig, W. A., Brewer, R. E., Holmes, C. R., Reynolds, D. A., and Sprunk, G. C., work cited in footnote 20, p. 10.

[81] Fieldner, A. C., Davis, J. D., Reynolds, D. A., Schmidt, L. D., Brewer, R. E., Sprunk, G. C., and Holmes, C. R., work cited in footnote 21, p. 10.

[82] Fieldner, A. C., Davis, J. D., Reynolds, D. A., Brewer, R. E., Sprunk, G. C., and Schmidt, L. D., Carbonizing Properties and Petrographic Composition of Lower Banner Bed Coal from Keen Mountain Mine, Buchanan County, Va., and the Effect of Blending This Coal with Pittsburgh Bed (Warden Mine) Coal: Bureau of Mines Tech. Paper 616 (In press).

[83] Fisher, C. H., Composition of Coal Tar and Light Oil: Fuel, vol. 18, May-October 1939, pp. 146-153, 171-179, 213-219, 249-254, 266-270, 305-310.

TABLE 4. - Physical properties of cokes carbonized at 900° C. in the 18-inch retort

Coal 55 - Sewell bed, Wyoming mine, Wyoming County, W. Va.
Coal 56 - Pocahontas No. 3 bed, Buckeye No. 3 mine, Wyoming County, W. Va.
Coal 57 - Pocahontas No. 4 bed, No. 4 mine, Raleigh County, W. Va.
Coal 58 - Lower Banner bed, Keen Mountain mine, Buchanan County, Va.
Coal 59 - Upper Freeport bed, No. 21 Mine, Monongalia County, W. Va.
Coal a36 - Alma bed, Red Jacket mine, Mingo County, W. Va.
Coal 28 - Pittsburgh bed, Warden mine, Allegheny County, Pa.

Coal No.[1]	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon - 2-in. screen	1 1/2-in. screen	1-in. screen	1/4-in. screen	Tumbler test, cumulative percent upon - 2-in. screen	1-1/2-in. screen	1-in. screen	1/4-in. screen
55	1.86	0.88	52.7	57.9	85.7	96.8	99.0	11.8	43.5	68.9	75.9
55A	1.86	.87	53.2	40.2	70.7	94.3	98.5	.9	22.1	61.2	75.1
55B	1.86	.86	53.8	40.4	75.0	94.0	98.6	6.1	30.8	63.6	77.3
56	1.86	.90	51.6	59.9	83.3	94.6	98.5	5.2	32.5	59.3	71.5
56A	1.89	.86	54.5	45.1	77.0	94.0	98.6	2.2	25.6	58.8	71.4
56B	1.87	.85	54.5	57.6	83.3	94.7	98.4	3.4	31.4	60.3	70.9
57	1.88	.91	51.6	62.2	87.3	95.2	98.3	9.0	37.4	60.7	70.6
57A	1.87	.85	54.5	46.3	79.5	94.7	98.3	3.0	31.4	60.3	71.8
57B	1.87	.85	54.5	51.3	84.1	95.2	98.7	3.2	33.9	61.8	72.8
58	1.91	.95	50.3	48.7	82.6	95.9	98.6	7.3	36.7	65.4	75.8
58A	1.86	.86	53.8	35.6	74.4	93.6	98.2	3.6	27.9	59.0	70.6
58B	1.87	.87	53.5	48.3	83.1	94.9	98.5	4.2	29.6	63.1	72.0
59	..	.81	..	47.2	82.4	94.1	98.5	3.6	32.7	58.9	69.0
a36[2]	1.84	.86	53.3	30.7	60.3	89.1	98.1	2.3	16.7	53.4	75.6
28[3]	1.85	.88	52.5	33.1	68.0	93.2	98.6	1.8	20.4	55.2	71.5

[1] Coal 55A contains 80 percent and coal 55B contains 70 percent of Alma-bed coal (no. a36); coals 56A, 57A, and 58A contain 80 percent and 56B, 57B, and 58B contain 70 percent of Pittsburgh-bed (Warden mine) coal (No. 28).

previously.[84], [85] Table 5 gives the sources and characteristics of the coals tested. Coals 54, 52, and 53 are high-volatile A, coals 58 and 55 medium-volatile, and coals 56 and 57 low-volatile bituminous in rank. Table 6 shows their relative rates of oxidation expressed on four different bases. Comparison of the data given in the two tables shows that the rate of oxidation in general decreases with decreasing volatile-matter content, or, more regularly, with the decreasing oxygen content of the fresh coals. In respect to storing qualities, it should be observed that the coals that oxidize the more rapidly also show lower friabilities, so that the one factor tends to compensate the other.

TABLE 5. - Sources and characteristics of coals tested

Coal No.	Bed	County	State	Ash- and moisture-free Volatile matter, percent	Ash- and moisture-free Oxygen, percent	Friability, percent	Agglutinating value kg.[1]
54	High Splint	Harlan	Ky.	39.9	8.8	14.4	2.6
52	Pittsburgh	Allegheny	Pa.	40.0	7.0	27.1	[2] 8.4
53	Pond Creek	Pike	Ky.	34.7	5.9	31.0	8.2
58	Lower Banner	Buchanan	Va.	24.0	2.8	56.6	5.3
55	Sewell	Wyoming	W. Va.	22.8	2.8	62.2	6.7
56	Pocahontas No. 3	do.	do.	18.9	2.3	68.4	5.1
57	Pocahontas No. 4	Raleigh	do.	18.5	2.6	..	6.2

[1] Crushing strength, in kilograms, with a ratio of silicon carbide to coal, 15:1.

[2] Crushing strength, in kilograms, with a ratio of sand to coal, 15:1.

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[84] Fieldner, A. C., work cited in footnote 24, p. 15.

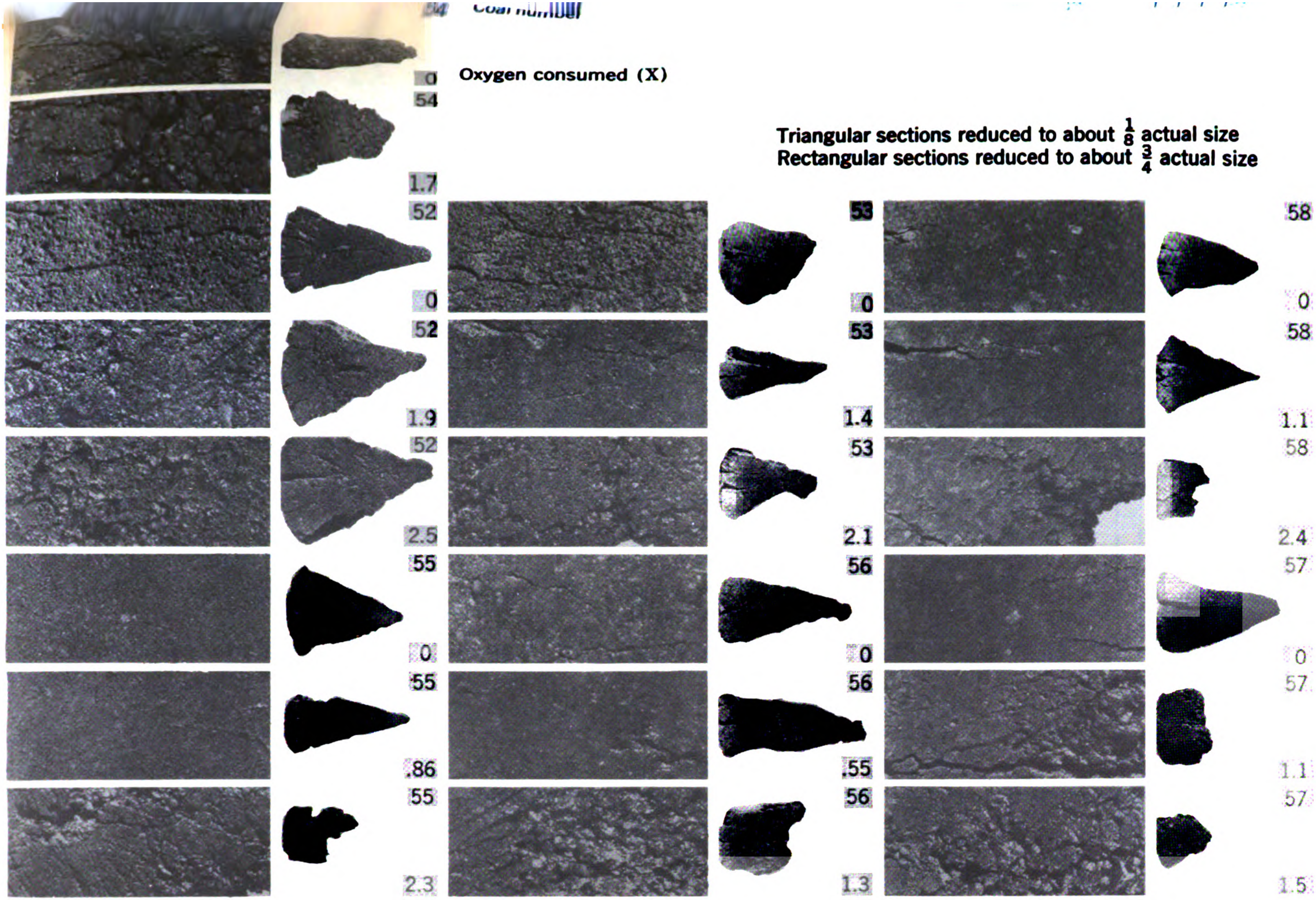
[85] Schmidt, L. D., and Elder, J. L., Atmospheric Oxidation of Coal at Moderate Temperatures. I. Rate of the Oxidation Reaction for Representative Coking Coals: Ind. and Eng. Chem., vol. 32, 1940, pp. 249-256.

TABLE 6. - Rate of oxidation of coal (minus 1/4-inch) in air at 99.3° C.

	Oxygen consumed						
Coal No.	54	52	53	58	55	56	57
At end of first day (percent by weight of dry, mineral-matter-free coal per day)	0.42	0.28	0.25	0.24	0.21	0.18	0.24
At end of first day (grams of oxygen per square meter of coal surface per day)	.29	.40	.34	.20	.19	.19	.28
After 1 percent oxygen has been consumed (percent of dry, mineral-matter-free coal per day)	.34	.21	.18	.17	.14	.12	.17
After 1 percent oxygen has been consumed (grams of oxygen per square meter of coal surface per day)	.47	.30	.24	.15	.13	.13	.20

Table 7 shows the effect of oxidation on the carbonizing properties of the seven coals in the BM-AGA 18-inch retort at 800° C. The carbonization-test results show that the amount of oxygen required to cause a 20-percent reduction in coke hardness ranges from 0.8 percent by weight of dry, minera matter-free coal for coal 57 (Pocahontas No. 4 bed) to 2.6 percent for coal 52 (Pittsburgh bed). The corresponding periods of oxidation of these coals in air (20.93 percent oxygen) at 99.3° C. before coking were 3.6 and 11.8 days, respectively. The results show further that the agglutinating value, yield of tar, and heating value can all be used as measures of the extent of preoxidation of the coal. In general, coke stability, shatter index, a extent of fusion vary in a manner similar to the hardness of the coke.

Figure 12 shows the effects of preoxidation of the seven coals upon their coking power. The lower figure in each photograph indicates the extent of oxidation, namely, the amount of oxygen consumed expressed as percent by weight of the coal (dry, mineral-matter-free basis). In this figur the sections that are roughly triangular show typical coke pieces (one-eighth actual size) as taken from the retort. The rectangular sections (three-fourths actual size) show the fine structure of the coke. The first effect of oxidation is to reduce the size of the cells or pores; further oxidation produces a pebbly coke because of inadequate fusion of the charg As indicated by the shape of the typical coke pieces, some coals were oxidized until, upon carbonization, only a 2-inch or 3-inch shell next to the heated wall of the retort was fused, the remainder of the retort being filled with granular coke. It is evident that this part of the charge did not become plastic during the coking process.



Triangular sections reduced to about $\frac{1}{8}$ actual size
Rectangular sections reduced to about $\frac{3}{4}$ actual size

Figure 12.- Photographs of coke sections showing effect of oxidation of coal.

TABLE 7. - Amount of oxygen required to cause 20 percent reduction in hardness of coke produced and corresponding effect on other carbonizing properties

Coal No.	54	52	53	58	55	56	57
Amount of oxygen required to cause 20 percent reduction in coke hardness (percent by weight of dry, mineral-matter-free coal)	1.4	2.6	2.2	2.1	1.8	1.1	0.8
Corresponding number of days oxidation in air at 99.3° C.	3.4	11.8	11.5	11.6	11.3	7.5	3.6
Corresponding percentage decrease in heating value of coal	.66	2.1	1.7	1.3	1.3	.79	.48
Corresponding agglutinating value (kg.)	1.6	6.1	4.9	3.4	3.2	3.8	4.2
Corresponding coke stability (percent on 1-inch screen after tumbler)	20	29	47	55	57	48	48
Corresponding coke stability (ratio to value for fresh coal)	.77	.84	.79	.78	.78	.76	.78
Corresponding coke shatter index (percent on 1-1/2-inch screen)	59	75	83	85	89	83	88
Corresponding shatter index (ratio to value for fresh coal)	.80	.98	.94	.90	.93	.88	.92
Corresponding tar yield (percent by weight of coal)	6.8	6.4	5.1	2.8	2.7	1.8	1.7
Corresponding tar yield (ratio to value for fresh coal)	.91	.66	.63	.72	.64	.75	.85
Corresponding percent unfused coke from retort (percent passing 1-inch screen)	29	2.2	3.8	25	18	30	22

Gas Pressures in Uncoked Portions of Charge During Progress of Coking

Gas pressures developed in the uncoked portions of the coal charge have been measured while coking is in progress in the cylindrical BM-AGA retort.[8 The manner in which these pressures develop is described. They increase irregularly as the plastic coal layer moves to the middle of the retort and reach a maximum at the time the innermost coal attains the plastic state. For the coals investigated, maximum pressures vary according to the rank of the coal, increasing with decrease of volatile matter in the coal. The maximum gas pressure developed in the carbonization of low-volatile coals is not greatly changed by the temperature of carbonization, provided the heating rate is rapid enough to insure a continuous plastic layer. The maximum pressures developed in carbonizing medium- and high-volatile coals decrease as the temperature of carbonization is increased. These pressures may contribute to the expansion of the charge by opposing the escape of the volatile matter from the layer of plastic coal inwardly to the zone of uncarbonized coal. It has been observed that with high pressures within the plastic seal the retorts are deformed. Figures 13 and 14 show the magnitude and variation of pressures for representative high-, medium-, and low-volatile coals (Pond Creek, Sewell, and Pocahontas No. 3, respectively) carbonized at 600° and 900° C., respectively. Figure 15 illustrates the extent of deformation of the retorts with low- and medium-volatile coals. The low-volatile coal deformed the retort 2.8 percent and the medium-volati 0.3 percent.

Determination of the Swelling Properties of Coals During the Coking Process

Apparatus and procedures used by the Bureau of Mines for determining the swelling or expanding properties of coals have been described.[87] The same test methods were used during the present year for determining the expanding properties of coals studied in the survey of carbonizing properties of American coals.[88] In addition, tests were made on seven coals submitted by Subcommittee XV of the American Society for Testing Materials Committee D-5.[89] The ranks, proximate analyses, and results of expansion tests in the sole-heated and slot ovens for the coals tested are given in table 8. The coals are arranged in the order of increasing rank. It is evident that coals of high rank generally expand more than those of lower

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[86] Reynolds, D. A., and Birge, G. W., Gas Pressures Within the Uncarbonize Part of a Coal Charge Undergoing Carbonization: Ind. and Eng. Chem., vol. 31, 1939. (In press.)

[87] Auvil, H. S., and Davis, J. D., Determination of the Swelling Properties of Coal During the Coking Process: Bureau of Mines Rept. of Invest gations 3403, 1938, 18 pp.; Fuel, vol. 17, November 1938, pp. 335-348.

[88] Auvil, H. S., Davis, J. D., and McCartney, J. T., Expansion of Coal During Coking: Bureau of Mines Rept. of Investigations 3451, 1939, 21 pp.

[89] American Society for Testing Materials, Report of Committee D-5 on Coa and Coke: Proc., vol. 38, pt. 1, 1938, pp. 408-411.

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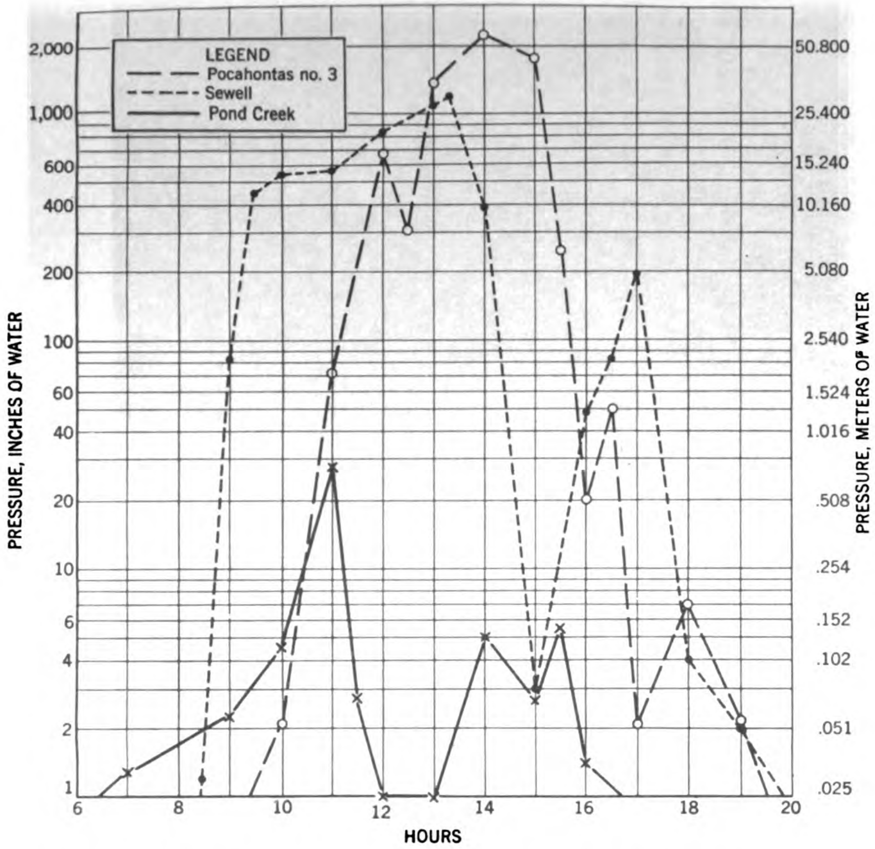

Figure 13.- Pressures developed by coals of varying rank at 600° C. in 33.0-cm. (13-in.) retort tests.

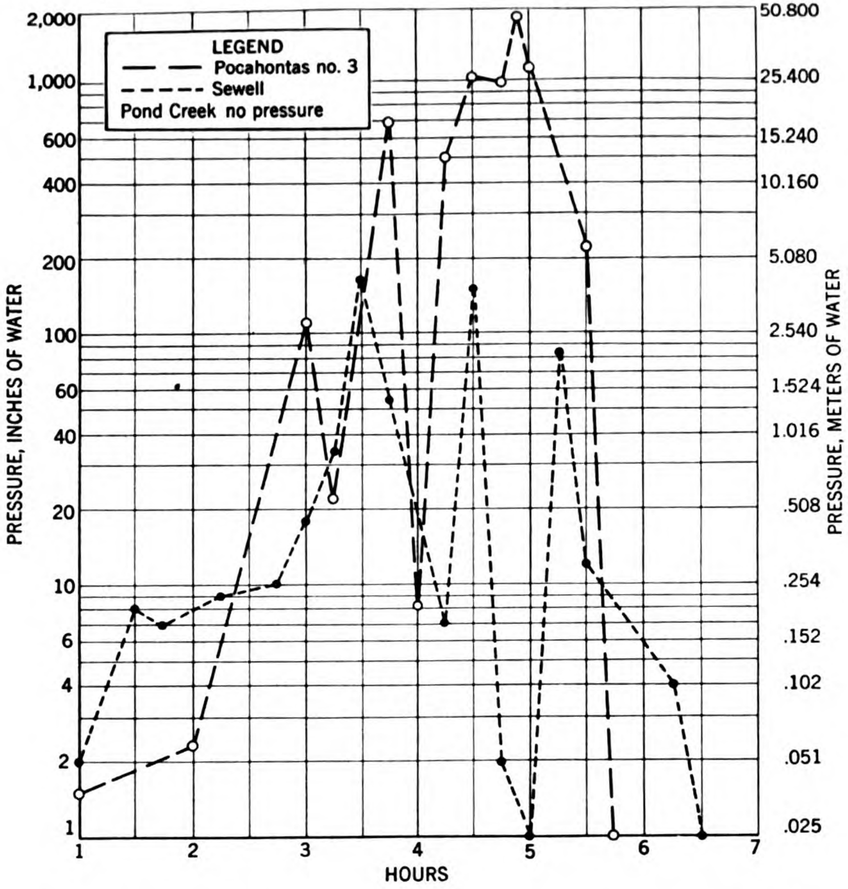

Figure 14.- Pressures developed by coals of varying rank at 900° C. in 33.0-cm. (13-in.) retort tests.

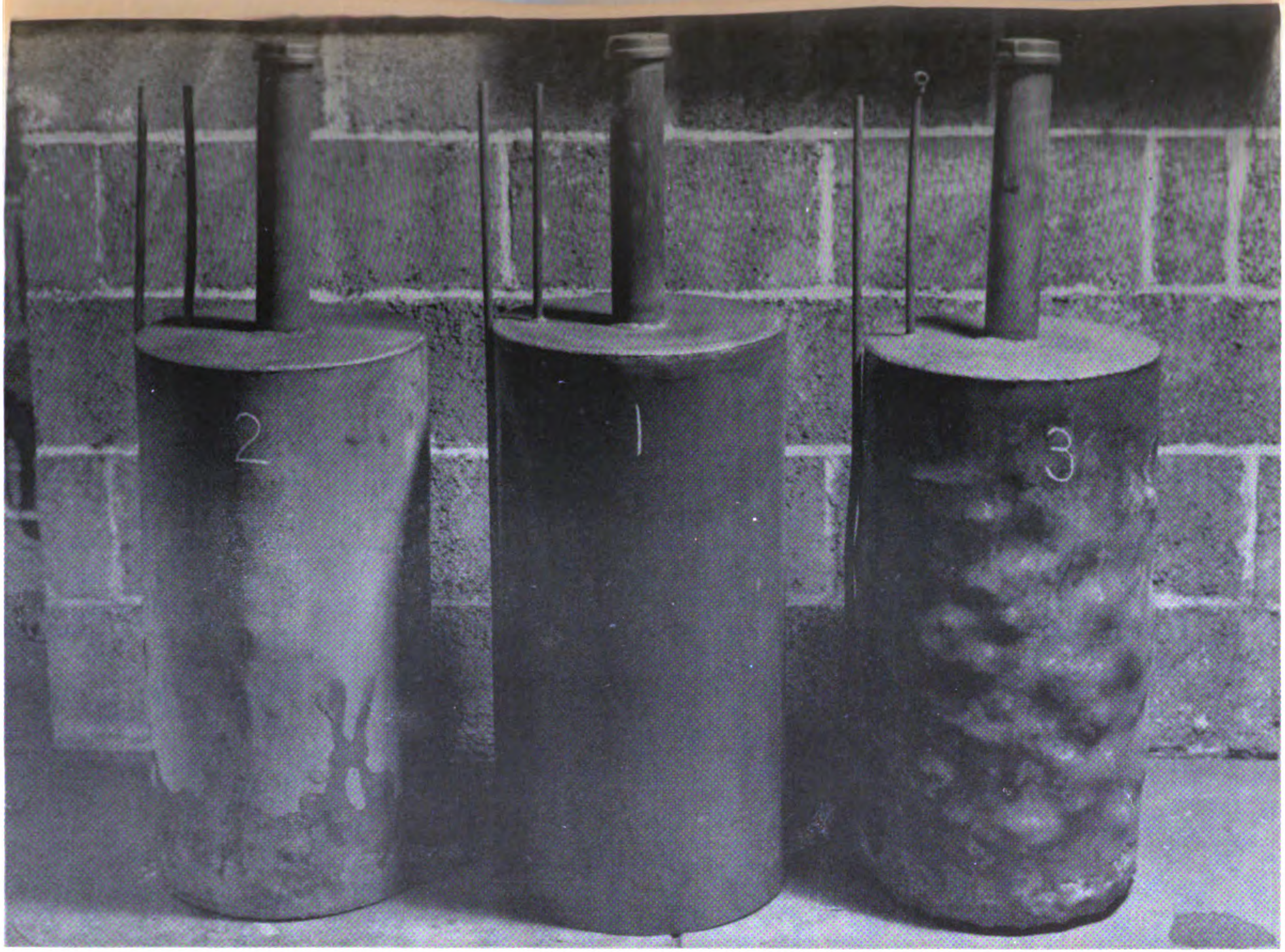

Figure 15.– Retorts deformed by expanding coals: (1) New retort; (2) retort moderately deformed by a medium-volatile coal; (3) retort greatly deformed by a low-volatile coal.

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rank, although the relation between rank and expanding properties is far from linear. It is indicated, therefore, that a rough idea of how coal will expand (or contract) in a coke oven can be gained from its rank, but that a close estimate can be obtained only by direct measurements of expansion. For example, it is generally considered that a coal that expands more than 2 percent under test should not be charged in a modern tall coke oven without a mixture of a contracting coal; table 8 shows that coals of virtually the same rank may differ more than 2 percent in expanding properties.

TABLE 8. - Analyses and expanding properties of coals
(Arranged in order of increasing fixed carbon)

Coal No.	Dry mineral-matter-free fixed carbon, percent	Proximate analysis, as carbonized basis, percent				Expansion at 55.5 pounds bulk density per cubic foot, percent	
		Moisture	Volatile matter	Fixed carbon	Ash	Sole-heated oven	Slot oven
XP-14	59.7	3.0	38.0	55.6	3.4	- 6.7	- 8.4
54	59.9	3.3	37.5	55.5	3.7	- 8.6	- 9.3
XP-12	60.4	7.5	33.2	48.8	10.5	- 7.7	- 8.0
d28	61.8	1.5	36.0	57.3	5.2	-13.7	-
XP-9	62.2	1.8	35.4	57.1	5.7	-15.3	-
XP-7	64.6	3.5	32.0	56.8	7.7	- 9.4	- 8.3
53	66.1	2.7	31.9	61.1	4.3	- 6.6	- 7.6
44	66.3	1.8	32.4	62.9	2.9	-	- 4.4
59	67.7	2.8	29.3	59.6	8.3	- 2.5	-
XP-13	73.2	1.8	25.8	69.6	2.8	+ 9.4	+13.4
60	73.5	2.0	24.9	66.7	6.4	+22.8	-
58	76.9	2.5	21.4	67.8	8.3	- 6.2	-
55	77.5	1.6	21.8	73.7	2.9	+11.7	+16.8
XP-10	78.8	1.1	20.4	74.0	4.5	+13.0	+16.3
XP-11	80.6	1.2	18.5	74.3	6.0	+12.3	+16.8
56	81.6	1.3	17.5	74.7	6.5	+17.8	+19.9
57	82.0	1.5	17.1	74.1	7.3	+22.6	+27.8
XP-8	82.8	1.1	16.5	75.6	6.8	+27.5	+30.9

No empirical test, such as those used by the Bureau of Mines to determine expanding properties, will give results applicable to all industrial coking processes, because carbonizing conditions vary for different processe and also in different installations of the same process. However, there are factors fundamental to carbonization, which, if varied either in the coke oven or the test apparatus, will affect expansion results; investigation of such factors is therefore a part of this study. The factors (or variables) studied so far include effect of rate of heating through the plastic range, effect of bulk density of the charge, and effect of shrinkage of the coke.

Figure 16 shows that, in general, increased rate of heating through the plastic range of temperature increases the expansion of expanding coals and the contraction of contracting coals. However, the magnitude of the effect varies for different coals.

Coal expands during coking only when it is in the plastic state. With granular coal ordinarily charged industrially, obviously the first expanding effect is the filling of voids between the granules with plastic coal. Any further tendency to expand after the voids are filled must cause externa expansion or side thrust on the walls of the oven, and this is the effect it is desired to evaluate. If there were no voids (as with a solid block of coal), the total force of expansion would be exerted on the walls. With these considerations as a basis, and knowing the external expansion at a given density of charge, it should be possible to calculate the expansion of solid coal or the external expansion at any other density of charge. If the sum of the voids in the charge and the external expansion is calculated as a fraction of the actual coal volume, a value is obtained that represents the actual expansion of solid coal. This value is designated as E_s, and the equation for calculating from test results is $E_s = V_s (1 + E_t) - 1$, where,

V_s = total volume of charge in cubic feet per cubic foot of solid coal at test conditions.

E_t = fractional expansion observed in the test.

Figure 17 shows test results obtained at various bulk densities for several coals plotted against expansion of the solid coal, as calculated by the formula. The graphs are approximately parallel to the abscissa, as the should be if the expansion of solid coal were independent of bulk density. The deviation from parallelism probably is due to variation of the conductivity of the charge with bulk density; this would affect somewhat the rate of heating through the plastic range, and it has been shown that expansion varies with this rate. It should be observed that all coals plotted in figure 19 expand in the solid state. Coal a52 is strongly contracting at normal densities of charging.

After the coke is formed, at about 500° C., further heating to higher temperatures, as in a coke oven, causes it to shrink, and this shrinkage diminishes the effective external expansion under practical coking conditions. Figure 18 shows the linear shrinkage of cokes from representative coking coals as a function of the maximum temperature of carbonization.

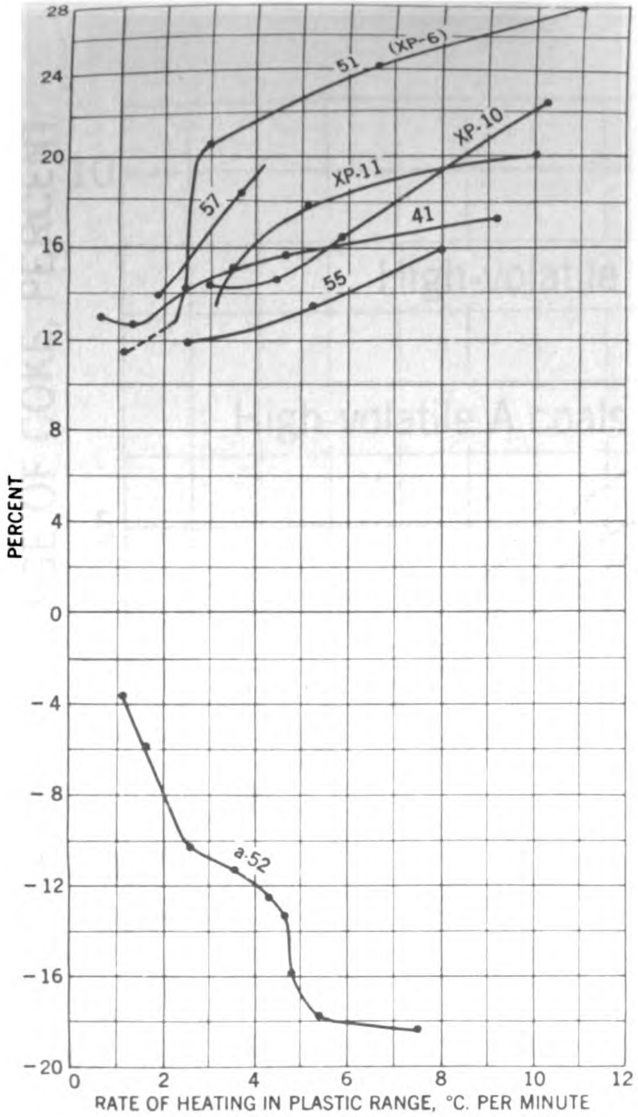


Figure 16.- Relation between expansion and rate of heating.

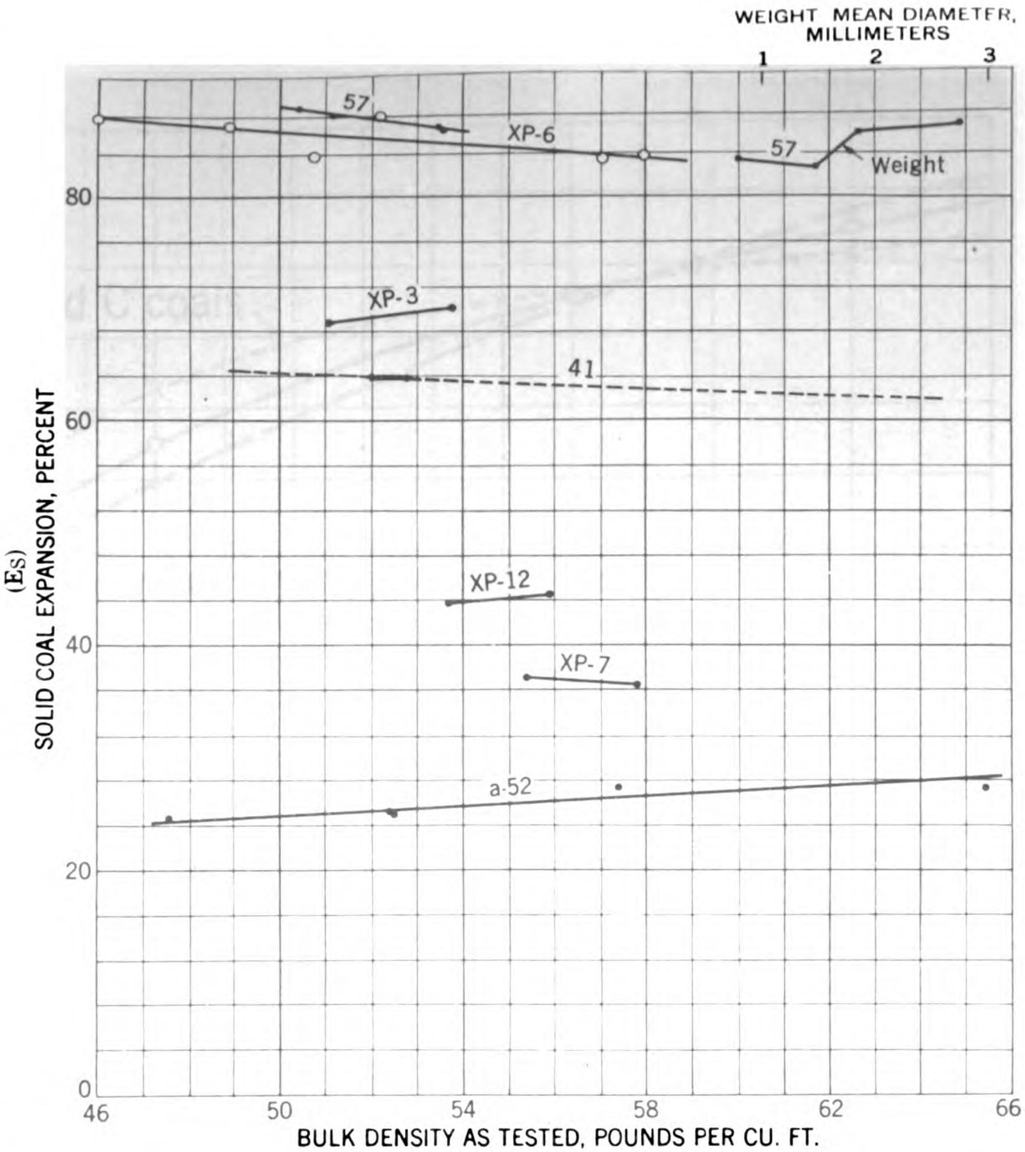


Figure 17.- Determined relation of solid-coal expansion to bulk density of charge.

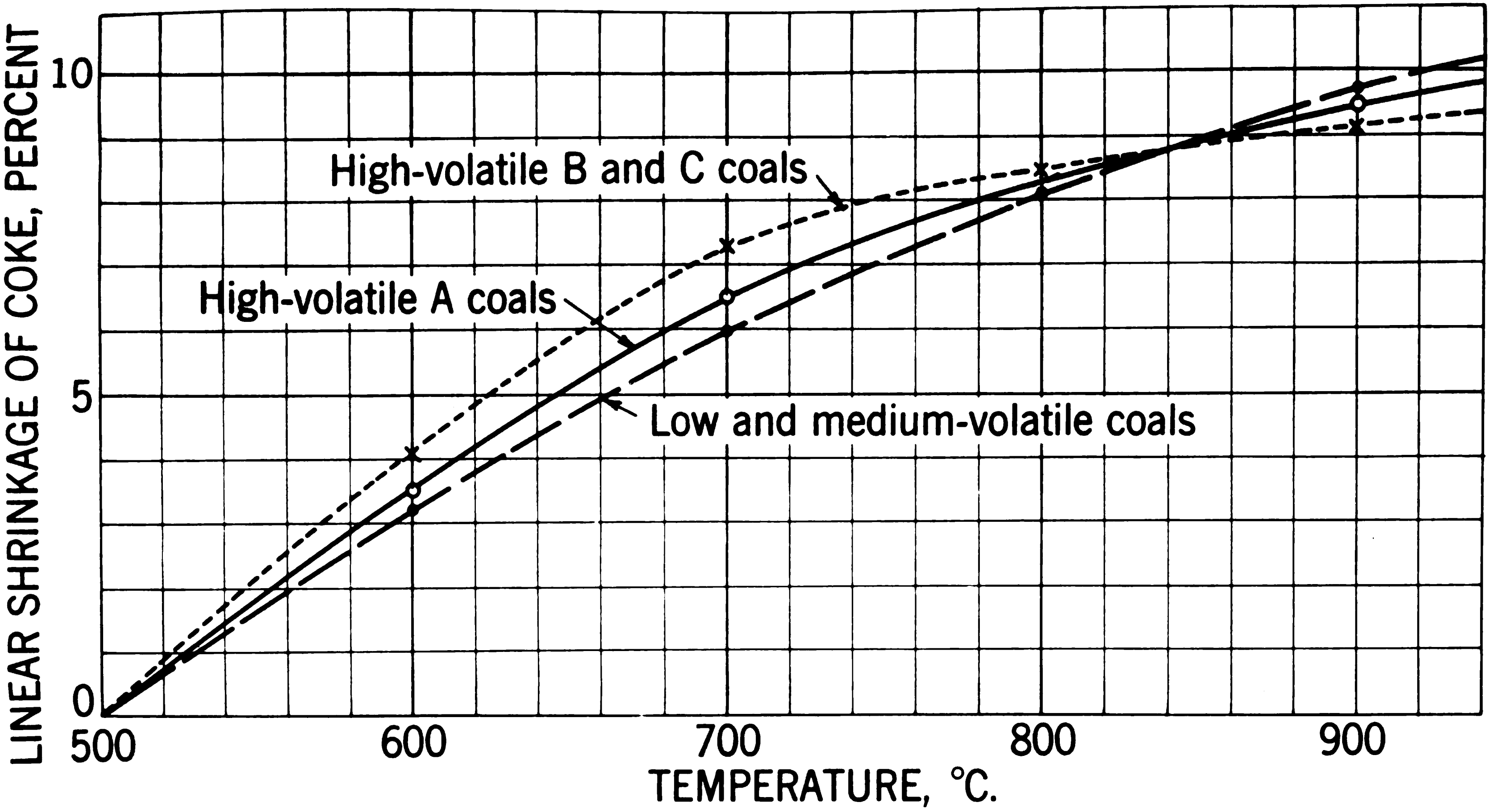
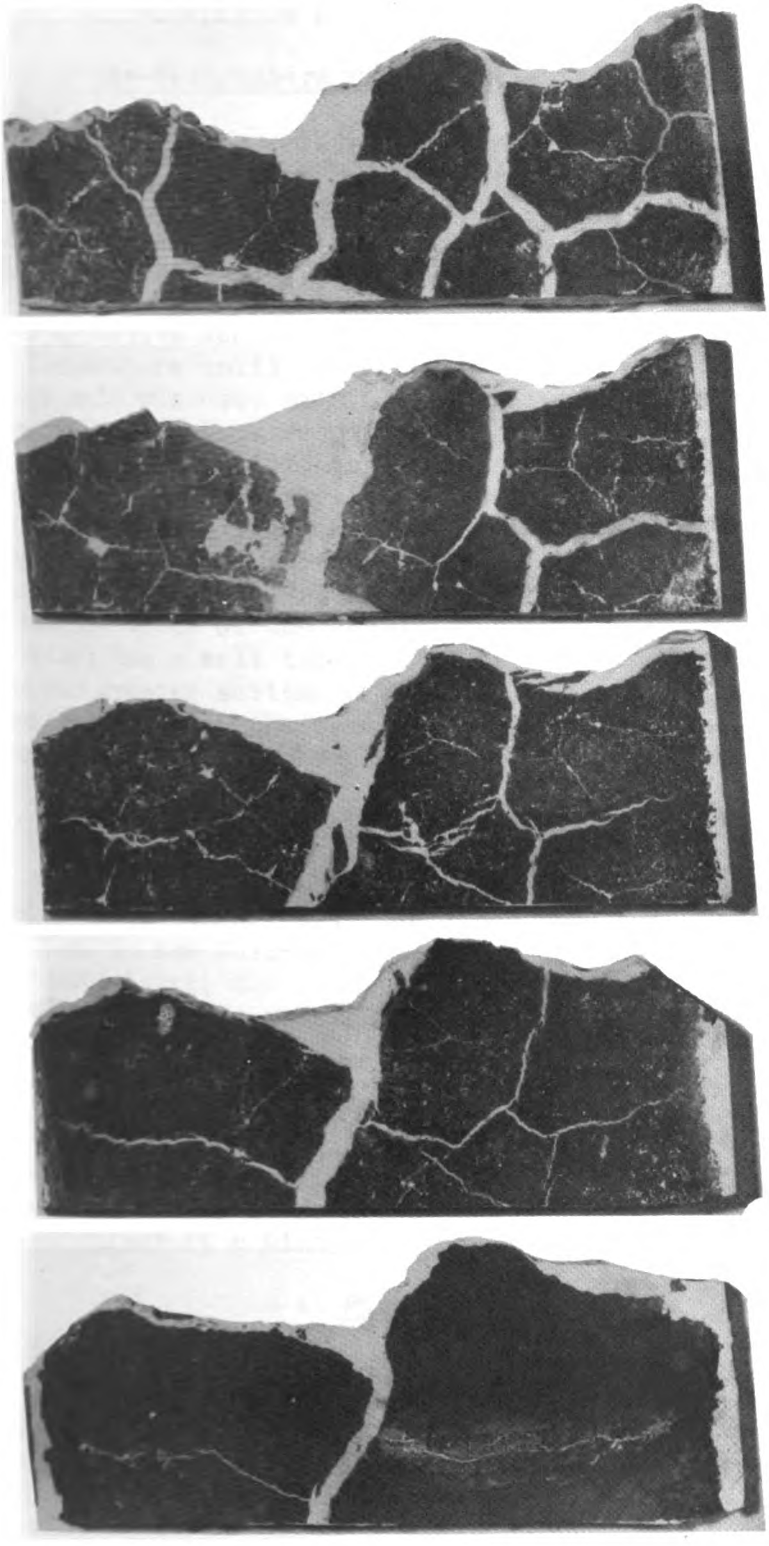


Figure 18.- Variation of the shrinkage of granular cokes with final temperature.



Distance from heated wall, inches	Maximum temperature of coke at section, °C.
0.67	945
1.80	895
2.94	815
3.92	695
4.55	575

Figure 19.- Cross sections of low-volatile coke from sole-heated oven.

Figure 19 shows the increase in shrinkage cracks in sections of coke as the maximum temperature of carbonization is increased.

Low-Temperature Carbonization of Subbituminous Coals

Low-temperature carbonization assays of a number of subbituminous coals from Colorado were made at various temperatures in three small retorts. These retorts included (1) a 50-gram Fischer assay retort, (2) a small, steel retort heated in the Fieldner electric furnace, and (3) a 5-pound, electric retort 4 inches in diameter.

Progressive heating to a predetermined temperature and maintenance of that temperature until the rate of evolution of gas was 20 percent of the average rate was used with all three types of retort tests. The Fischer assay and small steel retorts were heated to the maximum temperature in 1 hour; the 5-pound retort was heated at an average rate of 3.5° C. per minute. The latter test used sized coal, 1 to 1-1/2 inches. In this test, more than half of the gas formed in the early stages of decomposition is carbon dioxide, which first appears at about 400° C. and then decreases steadily until it is less than 2 percent in the gases formed at 700° C. The heating value of the gas and its rate of evolution reach maxima when the retort has a wall temperature of 600° C. The char shows a smaller yield and greater shrinkage up to 700° C. After 600° C. the char-breakage index remains about the same. The results by the Fischer low-temperature assays have been published.[90]

Carbonization of Subbituminous Coal in Curran-Knowles Coke Oven

Samples of 2- by 3- and 3- by 6-inch lump subbituminous coal from the Gorham Mine, Superior, Colo. were carbonized in test boxes under normal conditions in the Curran-Knowles sole-heated oven at West Frankfort, Ill. After carbonizing, the chars were quenched with steam and weighed, and the amounts of shrinkage within the boxes was determined. The chars were screened, submitted to a drop test of 3 feet on a concrete floor, and then rescreened. The chars were then shipped in sealed containers to Golden, Colo., and screen analyses were made to determine their degradation in transit. The chars were analyzed and their analyses compared with those of the two sized samples of the coal originally shipped to Illinois for carbonization. The study shows that the coals degrade to a smaller product when carbonized at a high temperature.

Hydrogenation of Coal

Temperature and pressure conditions for the hydrogenation of bituminous coal, the nature, importance, and industrial application of suitable catalysts to bring about the desired reactions, and the current outputs of various operating plants producing gasoline by coal hydrogenation have been summarized briefly.[91] This publication and earlier ones[92][93] describe the hydrogen-production and coal-liquefaction units of the Bureau of Mines experimental coal-hydrogenation plant and the results of a hydrogenation assay of Pittsburgh-bed, Bruceton-mine coal.

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[90] Parry, V. F., and Goodman, J. B., work cited in footnote 14, p. 9.

Data on Hydrogenation of Bruceton Coal

Data on the effect of varying temperature show that the yields of material soluble in benzene decrease and the losses as gas increase with increasing temperature.[94] The limiting low temperature is determined by the time of contact desired and the viscosity of the heavy-oil slurry, which is formed as a result of hydrogenation. From a practical point of view, the heavy-oil slurry must not be too viscous for convenient discharg at 100° C., and hence, if a lower temperature than 420° C. were desired, a longer contact time would be necessary. The ratio of overhead oil (the volatile oil that distills over the top of the converter) to heavy oil produced increases with increasing temperature. In operation of the experimental plant for assay purposes it was desirable to adjust the ratio of overhead to heavy oil so that the amount of the latter would exactly equal the weight of vehicle mixed with the original coal. With a mixture of about 40 percent coal (ash- and moisture-free) and 56 percent vehicle, the ratio of overhead to heavy oil for 70 percent oil yield should be 1 to 2. In addition to varying this ratio by changing the average reaction tempera ture, a very exact control may be exercised by regulating the amount of heat put into the top section of the converter, which in turn would contro the amount of refluxing. In the later runs with Bruceton coal, it was found essential to add to the heavy-oil slurry about 30 percent of its weight of overhead oil. This was necessary to make up for handling and sampling losses and to thin the tarry heavy-oil slurry so as to facilitate centrifuging and subsequent pumping of the paste made with the centrifuged oil. In a large plant, the handling losses would be virtually eliminated and the sampling losses would be negligible. It is probable, therefore, that the addition of 10 to 15 percent of middle oil (oil that distills at an intermediate temperature above that of the overhead oil) to the heavy-oil slurry will suffice to regenerate the pasting oil (oil that is mixed with pulverized coal before pumping the paste into the converters). This addition changes the ratio of overhead to heavy oil necessary for complete regeneration of pasting oil from 0.5 to about 0.85.

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[91] Storch, H. H., and Fieldner, A. C., Coal Hydrogenation. U. S. Bureau of Mines Experimental Plant: Mech. Eng., vol. 61, August 1939, pp. 605-611.

[92] Hirst, L. L., Hawk, C. O., Sprunk, G. C., Golden, P. L., Pinkel, I. I. Boyer, R. L., Schaeffer, J. R., Kallenberger, R. H., Hamilton, H. A., and Storch, H. H., Liquid-Phase Hydrogenation of Pittsburgh Seam Coal: Ind. and Eng. Chem., vol. 31, July 1939, pp. 869-877.

[93] Fieldner, A. C., work cited in footnote 24, p. 15.

[94] Hirst, L. L., and others, work cited in footnote 92, above.

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The result of increasing the contact time from 2 to 4 hours (decreasing rate of pumping paste from 7.5 to 3.8 pound per hour) at 440° C. is lower yields of oil and greater gas losses. For contact times less than about 1.6 hour (9.2 pound paste per hour) at 440° C., this trend reverses itself, so that shorter contact times result in lower yields of oil, although the gas losses are still decreasing. The exact location of the maximum yield cannot yet be stated with accuracy, but there is no question concerning its existence. The consumption of hydrogen increases with increasing temperature and time of contact.

No exact specifications can be given for optimum temperature and time of contact. Thus, there is a fairly wide range of temperature in which the time of contact may be adjusted so as to give the same yield of oil and the same viscosity of the heavy-oil slurry. This range of temperature for Bruceton coal is approximately 420° to 440° C.; the corresponding contact-time range is 2.75 to 1.75 hours. At temperatures higher than 440° C. a higher gas loss and lower yield of oil probably would persist, even at the limiting time of contact for obtaining a sufficiently fluid heavy-oil slurry.

A series of runs was made at 440° C., 2 hours contact time, 3,200 pounds per square inch pressure, and varying rates of recirculation of hydrogen from zero to 400 cubic feet per hour (measured at atmospheric pressure). These runs showed that the effect, if any, of increased recirculation of hydrogen was very small, that is, within the limits of reproducibility of the results. A similar conclusion has been reached by the British Fuel Research Station.[95]

Batch Hydrogenation in Small Autoclaves
Hydrogenation of Banded Constituents of Coal

To obtain more information on the relationship between type or nature of coal and its hydrogenation characteristics, the Bureau of Mines has prepared and hydrogenated about 70 samples of varied rank and petrographic composition. The details of the experimental work on these samples are given in publications now issued [96], [97], [98], [99] or soon to be published.[100], [101]

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[95] Great Britain Fuel Research Board, Report of the Fuel Research Board for the Year Ended 31st March 1937, with Report of the Director of Fuel Research: H.M.S. Office, London, 1937, p. 155.

[96] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., Hydrogenation of the Banded Constituents of Coal. Fusain: Ind. and Eng. Chem., vol. 31, February 1939, pp. 190-195.

[97] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., Hydrogenation of Spores, Resins, and Other Attrital Constituents of Coal: Fuel, vol. 18, May 1939, pp. 132-141.

[98] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., Hydrogenation of Anthraxylon from Bituminous Coals: Fuel, vol. 18, July 1939, pp. 196-203.

[99] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., Hydrogenation of the Banded Constituents of Coal. Attrital Matter and Anthraxylon: Ind. and Eng. Chem., vol. 31, September 1939, pp. 1155-1161.

The samples were carefully selected by hand, and their petrographic composition and purity were determined in all instances by microscopic examination of thin sections mounted on slides. Seven samples of fusain ranging from 88 to 93 percent carbon and 0.3 to 0.4 percent oxygen were separated by hand from 0.3- to 0.5-cm. layers in the coal. Pure spores from a Michigan high-ash coal, commercial lycopodium spores, and a spore-rich cannel coal were studied. Information regarding the hydrogenation behavior of oil algae was obtained with a boghead cannel coal containing a large proportion of this material. Pure resins, one sample from a Utah coal and one from a Canadian coal, both of the extruded type, were hydrogenated. Thirteen of the attrital samples contained considerable amounts of translucent attritus, whereas 13 others had large amounts of opaque matter. The anthraxylons included 14 samples from bituminous coals of eastern North America and 18 samples taken from peat, brown coal, lignites, and subbituminous coa of the western United States. Some of the bituminous and most of the low-rank anthraxylons contained considerable amounts of cellular resin.

To obtain comparable results, nearly all the experiments were made under the following standard conditions: One hundred grams of coal pulverized to pass through a 200-mesh sieve, 100 grams of tetrahydronaphthalene, and 1 gram of stannous sulfide were placed in a 1.2-liter 18 Cr-8Ni steel autoclave. Air was flushed out with nitrogen and hydrogen; then hydrogen was added until the pressure was 1,000 pounds per square inch at 20° C. The temperature was brought to 430° C. within about 1.5 hours and maintaine there for 3 hours. The autoclave was rotated on its longitudinal axis to provide agitation. Although more drastic conditions undoubtedly would have given higher yields with many of the samples, it was preferred to work wit these standard conditions because it was believed that comparatively mild conditions would be superior in revealing differences in hydrogenation behavior of the various constituents.

Owing to differences in quality and quantity of mineral matter in the samples, the conditions of hydrogenation were not always identical. For example, the mineral matter in the various samples might have exerted defi ite catalytic or anticatalytic effects. Moreover, the ratio of carbonaceo matter to vehicle and hydrogen varied somewhat with ash and moisture conte However, these differences are of minor importance for comparisons of the amenability of the various coal constituents to hydrogenation.

Since the liquefaction of spores was virtually complete at 400° C., the standard temperature of 430° C. was not employed here. It was observe

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[100] Eisner, A., Sprunk, G. C., Clarke, L., Fisher, C. H., and Storch, H. Hydrogenation of Typical Splint Coals: Ind. and Eng. Chem., vol. 32, 1940, pp. 73-78.

[101] Eisner, A., Sprunk, G. C., Clarke, L., Fein, M. L., Fisher, C. H., an Storch, H. H., Hydrogenation and Petrography of Some Low-Rank Coals f Western United States: Bureau of Mines Rept. of Investigations, 1940 (In press).

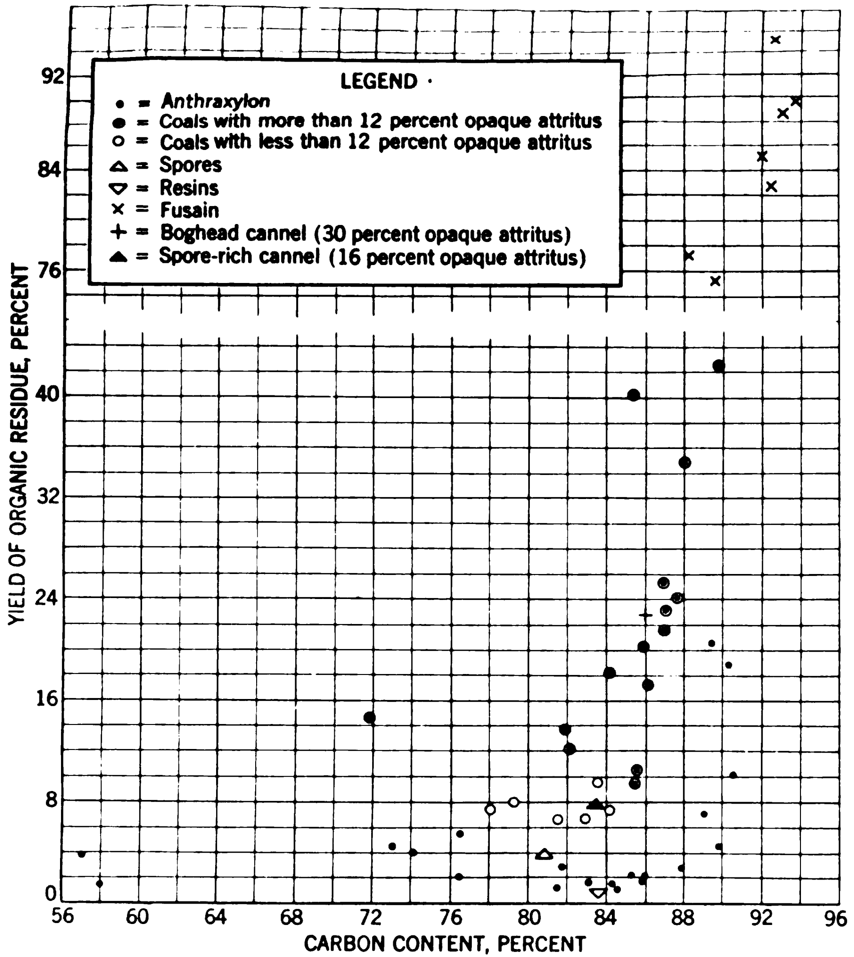

Figure 20.- Yields of organic residue and carbon content of sample hydrogenated (dry, ash-free basis).

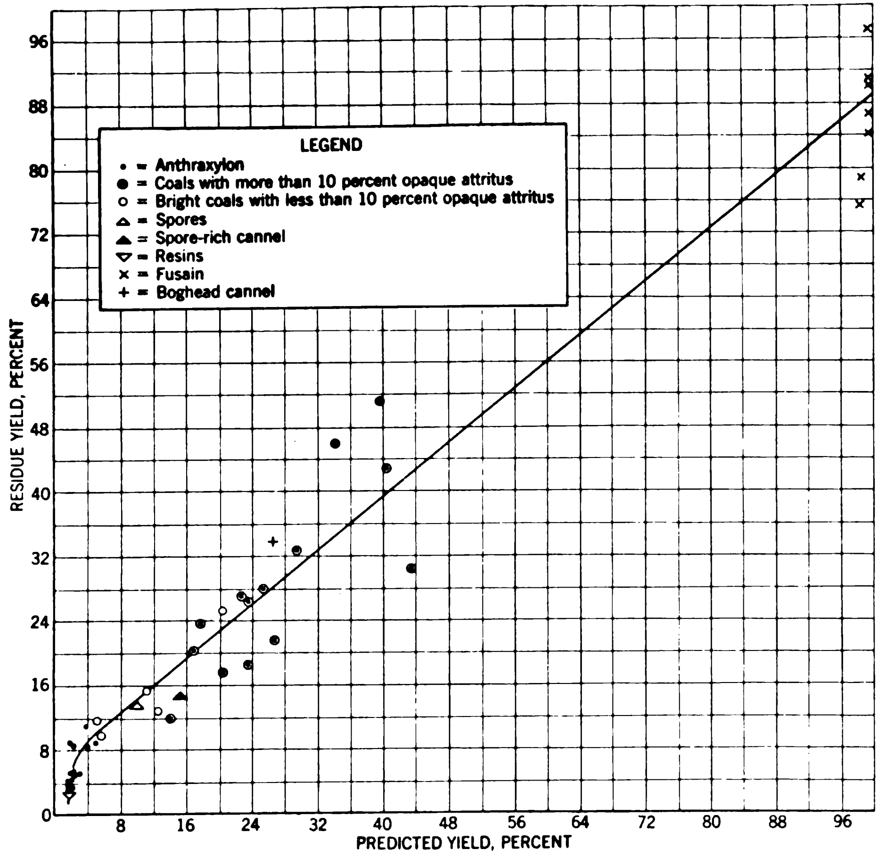

Figure 21.- Yields of residue predicted from petrography.

that several anthraxylons also gave excellent yields at 400° C. Several samples that gave low liquefaction yields under standard conditions were hydrogenated under more favorable conditions. Some of the high-rank anthraxylon and splint samples were hydrogenated at 440° C. for 3 hours with an initial (cold) hydrogen pressure of 1,800 pounds per square inch. A number of the low-rank anthraxylons that contained considerable amounts of cellular resins were observed to give higher liquefaction yields at 400° C. with an increased contact time (6 to 9 hours).

Details of the methods used in the separation and analysis of the liquid and solid products have been published.[102], [103] Hydrogenation yields were calculated from the yields of insoluble residue. Since small manipulative losses of the residue undoubtedly occurred, the yields reported are 2 or 3 percent high. These yields do not represent yields of oil, since, as is especially true for high-oxygen coals, water, carbon dioxide, gaseous hydro-carbons, ammonia, and hydrogen sulfide are formed, and their total is an appreciable fraction of the "liquefaction" yield. Although the criterion of liquefaction is arbitrary, since it is determined by the amount of residue insoluble in tetrahydronaphthalene, acetone, and benzol it is, nevertheless, satisfactory for the purposes of semiquantitative comparisons.

The results obtained with fusain, with anthraxylons (vitrains) of different ranks, and with the different components of the attritus (spores, resins, oil algae, translucent attritus, and opaque matter) are given in detail in the several papers cited.[104], [105], [106], [107] The last paper gives a comparative summary of the liquefaction yields. Figure 20, taken from this paper, shows the insoluble organic residue plotted against carbon content (dry, ash-free), and thus gives a comparison of data obtained on samples listed in the legend of this figure. The liquefaction yields (100 minus the yield of insoluble organic residue) with these samples show that appreciable liquefaction was effected in all cases. All of the seven samples of fusain were liquefied to an appreciable extent; two to about 25 percent. All of the samples of anthraxylon containing less than 89 percent carbon were liquefied in excellent yield - above 94 percent; those with higher carbon gave lower yields. Coals with more than 12 percent opaque attritus, most of them with 82 percent or more of carbon, showed liquefaction yields up to 90 percent; coals with less than 12 percent opaque attritus gave liquefaction yields of more than 90 percent. Spores, resins, and the spore-rich cannel (16 percent opaque attritus) all gave liquefaction yields above 92 percent; the boghead cannel coal (30 percent opaque attritus gave 77 percent.

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[102] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., Storch, H. H., work cited in footnote 96, p. 53.

[103] Fein, M. L., Eisner, A., Cooper, H. M., and Fisher, C. H., Solubility Characteristics of Tars and Pitches Produced by Coal Hydrogenation. Determination of Insoluble Matter: Ind. and Eng. Chem., Anal. Ed., vol. 11, August 15, 1939, pp. 432-438.

[104] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., work cited in footnote 96, p. 53.

[105] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., work cited in footnote 97, p. 53.

[106] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., work cited in footnote 98, p. 53

[107] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., work cited in footnote 99, p. 53.

The summary just given serves only in a general way as a satisfactory means of predicting the liquefaction yield to be expected from any sample whose carbon content is known. Reference to the original paper[108] will show that the liquefaction yields from individual samples are affected greatly by the type and composition of the material present and by the experimental conditions of hydrogenation. Changes from "standard" to a special set of conditions more favorable for the individual sample may result in different yields.

Present State of Correlation between Petrography and Liquefaction Yields

Coals frequently have been classified as suitable or unsuitable for hydrogenation on the basis of carbon content. The success that might be expected from predicting yields of residue on the basis of carbon content alone is illustrated by figure 24. Apparently there is a rough correlation between carbon content and yield within the petrographic groups. It is interesting to note that there is also a rough relationship between the carbon and opaque attritus contents of these splint samples. Fusains and anthraxylons of the same carbon contents (about 90 percent) would be expected, from figure 24, to give liquefaction yields of about 20 and 90 percent, respectively. Smaller differences in yield would be expected from anthraxylons and splint coals of equal carbon content.

It is interesting to consider the facts responsible for the partial success experienced in correlating hydrogenation yields with carbon content of the sample, usually a bright coal. For relatively homogeneous coals (bright coals), the carbon content, carbon-hydrogen ratio, volatile-matter content, etc., are fair indexes of rank or degree of coalification. Since amenability to liquefaction decreases with increase in rank and virtually disappears for carbonaceous matter of very high carbon content (fusinite), the importance of carbon content in predicting yields is easily understood The situation is much more complicated with the chemically hetrogeneous du or splint coals. For such coals the carbon content, being only an average value, may be deceptive and, owing to the influence of spores, resins, etc. may not reveal the presence of considerable amounts of fusain and opaque attritus. The carbon-hydrogen ratio and volatile-matter content also are not useful since they, too, are average values. Petrographic methods, however, are especially valuable for heterogeneous coals, since a quantita petrographic estimate is possible of the constituents that differ widely hydrogenation behavior.

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[108] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. work cited in footnote 99, p. 53.

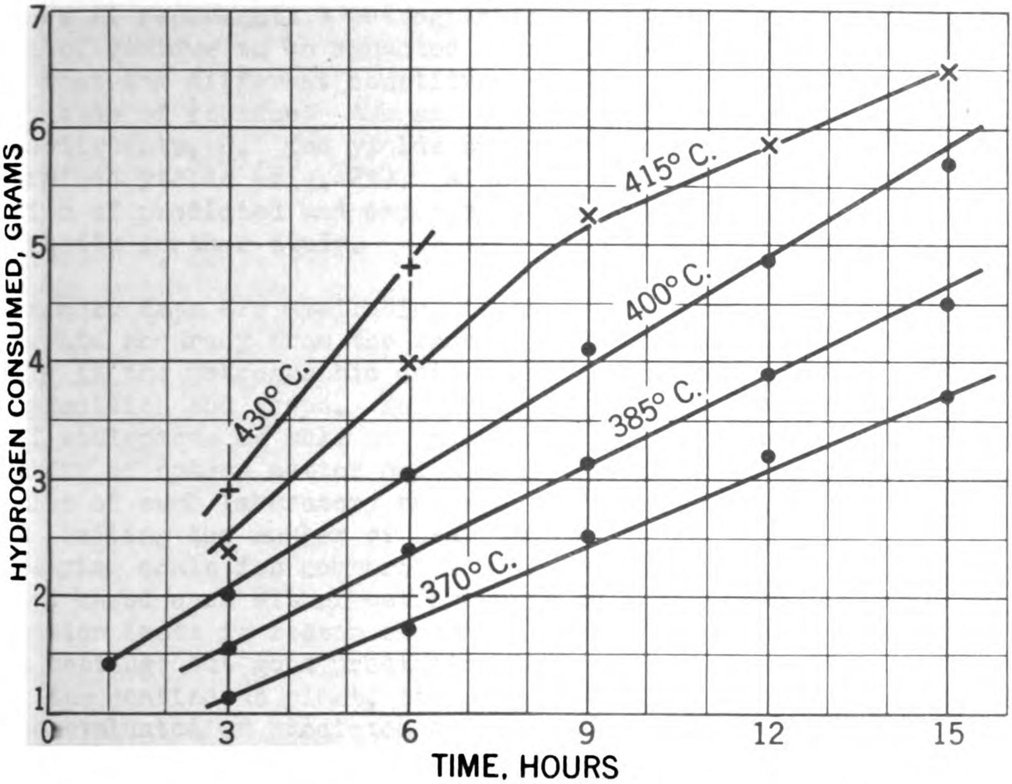

Figure 22.- Rate of hydrogen consumption at various temperatures.

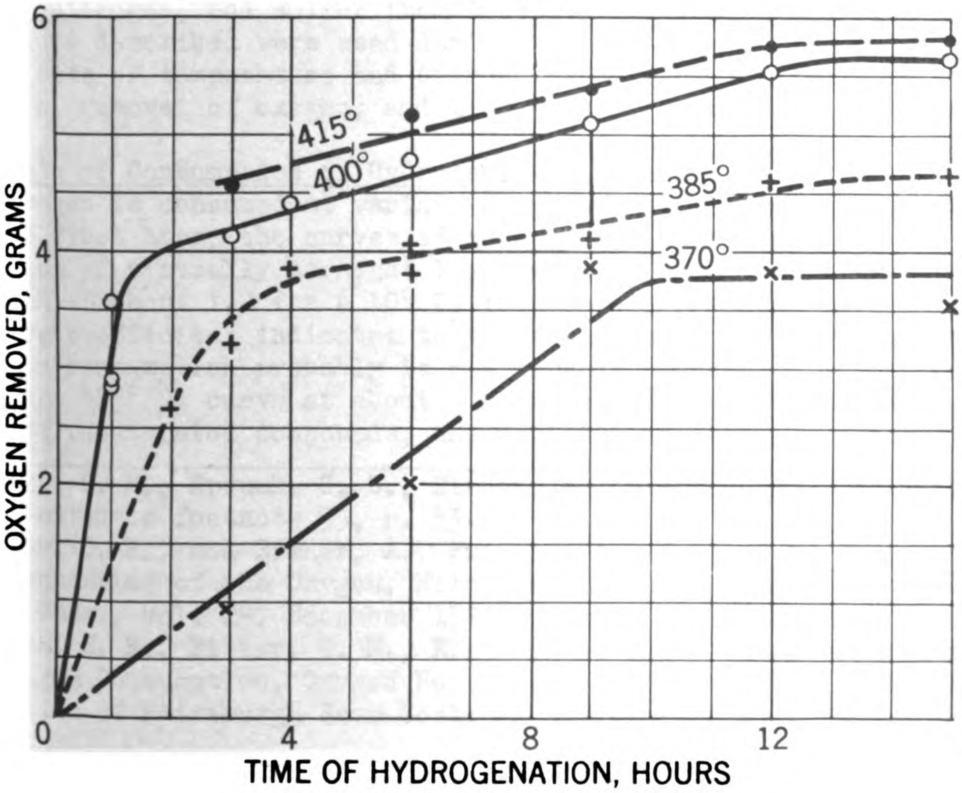

Figure 23.- Effect of time on oxygen removal.

Figure 21 represents a petrographic method of predicting yields.[109] The yield of residue to be expected from the petrography was estimated by assuming that the different constituents would give the following percentage yields of residue: Ash and fusain, 100; opaque attritus, 38; all other constituents, 0. The yields predicted in this way are plotted against the determined yields (fig. 21). Although far from satisfactory, this correlation of predicted and experimentally determined yields is interesting and merits further study.

When more data are available, it should be possible to predict yields with moderate accuracy from the rank and petrographic analysis of the coal, especially if the petrographic determinations can be improved with respect to both precision and scope. For example, it would be helpful to know the amount of semiopaque as well as opaque matter, or, better still, to have the quantity of opaque matter and a numerical index of its average opacity. The results of such laboratory analyses and tests should be of considerable value in limiting the number of coal samples to be tested in a continuous plant assaying coals for commercial hydrogenation. A poor liquefaction prediction based upon either petrographic analyses or small autoclave hydrogenation tests is reason enough to reject a coal for continuous hydrogenation testing; but good prediction does not preclude the necessity of tests in the continuous plant, since physical factors are involved that cannot be evaluated or predicted by petrographic or small autoclave tests.

Rates and Mechanisms of Coal-Hydrogenation Reactions

Some preliminary data have been reported on the rates of elimination of oxygen, nitrogen, and sulfur from Bruceton coal.[110] The same apparatus and procedure described were used during the present year in obtaining data on the effects of temperature and of rank of coal on the rates of consumption of hydrogen, removal of oxygen, and liquefaction of coal.[111]

A. Rate of Consumption of Hydrogen. - Figure 22 shows the rate at which hydrogen is consumed at various temperatures. After a steep rise during the first hour, the curves of consumption of hydrogen versus time are a series of virtually straight lines whose slopes show a temperature coefficient of about 1.2 for a 10° C. temperature change. This rather low temperature coefficient indicates that the slow step involved in the rate of hydrogen consumption probably is a diffusion process. The change in slope of the 415° C. curve at about 9 hours may be due to reversible hydrogenation of unsaturated compounds, the equilibrium favoring saturation at

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[109] Fisher, C. H., Sprunk, G. C., Eisner, A., Clarke, L., and Storch, H. H., work cited in footnote 99, p. 53.

[110] Fisher, C. H., and Eisner, A., Primary Liquefaction by Destructive Hydrogenation of the Oxygen, Nitrogen, and Sulfur Linkages: Ind. and Eng. Chem., vol. 29, December 1937, pp. 1371-1376.

[111] Storch, H. H., Fisher, C. H., Eisner, A., and Clarke, L., Kinetics of Hydrogen Consumption, Oxygen Removal, and Liquefaction in the Hydrogenation of Pittsburgh Seam Coal: Fuel, (In press.)

400° C. but changing rapidly at higher temperatures in favor of the unsaturated molecules. The steep rise during the first hour for all of the curves of figure 22 may be due to the initial solution of hydrogen in the liquid phase necessary to reach the steady state involved in the diffusion process. The latter may be the diffusion of dissolved hydrogen through a film covering the catalyst surfaces.

B. Rate of Removal of Oxygen. - Figure 23 shows the rates at which oxygen is removed at various temperatures. The rather abrupt changes in slope of these curves at about 4 hours, or 60 percent total oxygen removal from the coal, might suggest a rapid first-order, thermal reaction followed by a zero-order catalytic process that is obscured at temperatures higher than 400° C. by some secondary reaction that retards further elimina tion of oxygen; however, a series of successive unimolecular reactions migh give the types of curves shown in figure 23.

Detailed analysis of the oxygen-removal curves shows that the initial reaction for temperatures above 370° C. is a decomposition of the coal substance involving the elimination of oxygen. Except for the 385°-400° C. interval, the over all reaction that follows the initial thermal decomposition has a negative temperature coefficient. The negative temperature coefficient for the 370°-385° C. interval may be due to the fact that at 370° C. the initial reaction has not yet been completed at 8 hours reactio time, and hence comparison of the rates at 370° C. with those at 385° C. is not valid at 8 hours; even at 12 hours the very low positive coefficien indicates that the initial reaction is not yet completed at 370° C. Between 385° and 400° the positive temperature coefficient of a second reaction or group of reactions appears. These reactions may be catalytic in nature, involving adsorption of the reactants on a solid surface. Between 400° and 415° C. this second reaction is largely obscured by condensation reactions, which result in the production of molecules that are more stabl than the original coal substance and from which oxygen is eliminated at a very slow rate.

C. Rate of Liquefaction. - "Liquefaction" was measured by determinin the amount of residue obtained upon centrifuging the tetrahydronaphthalen solution of the product and subsequently washing the centrifuge residue with acetone and benzene. It is apparent, therefore, that by the term "liquefaction" is meant decrease in molecular weight until the products ar gaseous under normal conditions or dissolved by the treatment described above. Any further decrease in molecular weight will not be evaluated by the percent liquefaction.

Figure 24 shows the percentage of conversion of dry, ash-free coal into soluble material plus gases at various times and temperatures. Sinc the extent of liquefaction is independent of the rate of consumption of hydrogen and since, in most instances, virtually all of the tetrahydronaphthalene was recovered as such, the rate of liquefaction may be considered to depend only on the concentration of the coal substance.

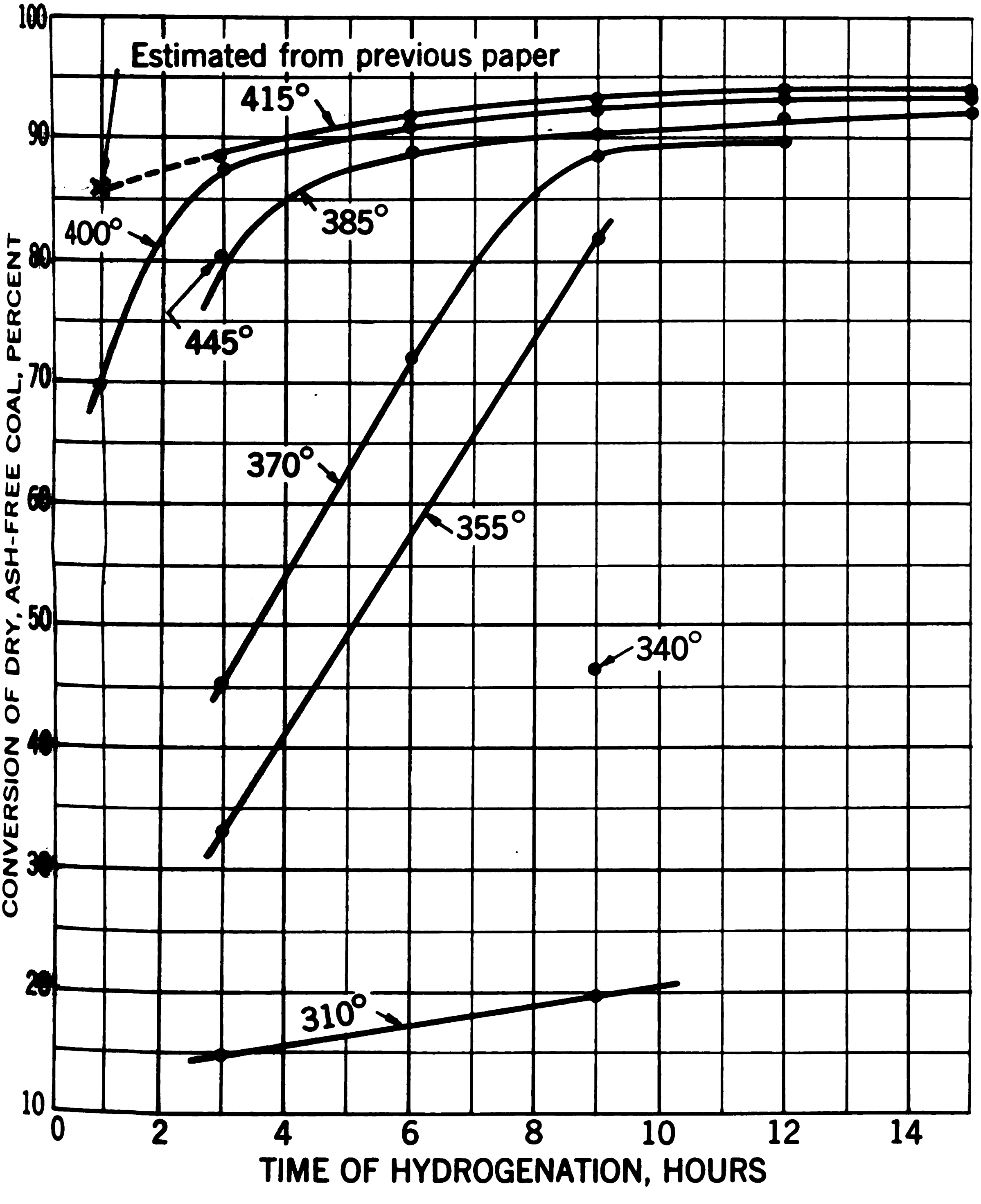

Figure 24.- Effect of time on liquefaction yield.

It appears from an analysis of the rates that at least three processes are involved in the liquefaction process, namely:

1. Solution or extraction of the coal substance. This is characterized by a low-temperature coefficient of about 1.2 per 10° C. (1.5 per 15° C.), and by the fact that it appears to be the principal process ocurring at temperatures below 370° C. The limiting factor in this process is probably (as in most solution processes) the diffusion of the dissolved material away from the solute surfaces.

2. Beyond 370° C. the temperature coefficient for the initial reaction increases rapidly to about 2.0 per 10° C.; the activation energy remaining at 61 (± 5) kg.-cal. in the temperature range 370°-415° C. This reaction is probably a thermal decomposition of the coal substance identical with the primary reaction involved in the elimination of oxygen and involving the rupture of carbon-to-oxygen bonds. Immediately after the production of free radicals or unsaturated molecules by the thermal decomposition reaction, most of these are saturated by catalytic hydrogenation. Those that are not stabilized by hydrogenation or by intramolecular rearrangement combine with each other and form a polymer more stable than the original coal substance.

3. The apparent negative temperature coefficients that are found at 8 and 12 hours for temperatures above 370° C. may be caused by the fact that the "coal substance" is not a chemically pure material and that its constituents (anthraxylon, translucent attritus, and opaque attritus) have different first-order rate constants. Another possible explanation of these negative temperature coefficients is the precipitation of polymers formed by the condensation products of the initial thermal decomposition of the coal. These polymers are more stable than the original coal, hence the rate constants for their liquefaction would be smaller than for the original coal. At a temperature of about 440° C. and 180 atmospheres pressure of hydrogen, the rate of precipitation of these polymers exceeds the rate of their liquefaction, hence increased amounts of residue appear, which are commonly designated "coke".

The absence of a positive temperature coefficient in the 385°-400° C. interval at 8 and 12 hours distinguishes the liquefaction data from those for elimination of oxygen. A possible explanation of this is that the condensation products formed in this temperature interval are fairly reactive so far as elimination of oxygen is concerned, but their liquefaction rate is enough smaller than that of the original coal to explain the negative coefficient.

The characteristic differences in the slopes of the curves in figures 22, 23, and 24 show that there is no direct or simple relationship between rate of consumption of hydrogen and rates of removal of oxygen and of liquefaction of coal.